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FINAL REPORT NO. 137

ITEM NO. 22

CYCLOPOLYOLEFINES

(Paper by Dr. J. W. Reppe, I. G. Farben Research Chemist)

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

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CYCLOPOLYOLEFINES

(Paper by Dr. J.W. Reppe, I.G. Farben Research Chemist).

Translation issued by
B.I.O.S. Group 3

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INTRODUCTION TO A TRANSLATION OF A PAPER BY DR. REPPE ON CYCLOPOLYOLEFINES

This report is a translation of a paper by Dr. Julius Walter Reppe. It was obtained by a C.I.O.S. CAFT Assessment Team (Major A.J. Skey, C.M.H.Q. and Mr. K.T. Nilsson, U.S.) during a visit in May last to the Anorgana G.m.b.H. plant at Gendorf, Bavaria. It describes the main work on Cyclopolyolefines carried out during the war under Reppe's direction at the IG plant in Ludwigshafen. Reppe was chief of the Organic Section of the main laboratory (Hauptlaboratorium) up until the latter stages of the war, when he moved with part of his staff to Gendorf.

Reppe was very interested in this work, and considered that he had opened up an important new field, one which was not only of academic interest, but which also held considerable promise industrially. This view was also shared by some of the Directors of IG, and were it not for the war they would have pressed forward with further research and development.

Various factors have contributed towards the delay in production of this translation, not the least being the fact that the original document went astray in transit. In view of its very great chemical interest, however, it is now submitted for reproduction and general release as a B.I.O.S. report.

A.E. CHILDS
Chairman, Group 3 B.I.O.S.
29th November, 1945.

Translation of a paper by Dr. Walter Reppe

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Ludwigshaven, 28.4.1944.

Cyclopolyolefines

A. Introduction.

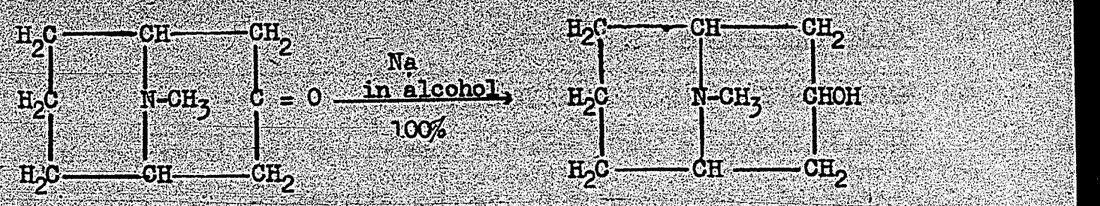
Through Berthelot's classic work it is known that acetylene enters into pyrogenetic condensation (at a high temperature) with the formation of aromatics. Berthelot 1) was able to obtain diphenyl, naphthalene and other aromatic hydrocarbons, besides benzene by means of the pyrogenic treatment of acetylene. Following Berthelot, a great many scientists have dealt with the tendency to polymerization of acetylene and the possibility offered thereby of obtaining valuable compounds synthetically. Papers by R. Meyer 2) and N. Zelinski 3) should be mentioned and one recently by R. Schwarz 4) who achieved the pyrogenic condensation of acetylene in a quenching tube, obtaining increased amounts of benzene and toluene. By a basically different method, Nieuwland, starting directly from acetylene, succeeded in producing its aliphatic polymerides, vinyl-acetylene, divinyl-acetylene, ethenyl-butadiene and the like.

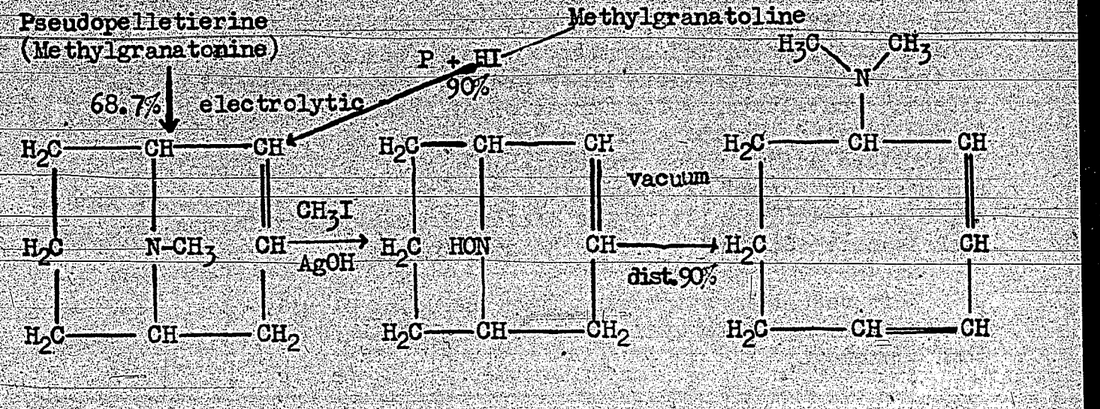
Our own investigations into the acetylene field reveals the astonishing fact that it is possible, by employing certain catalysts, to condense four or even more acetylene molecules to give cyclic polyenes.

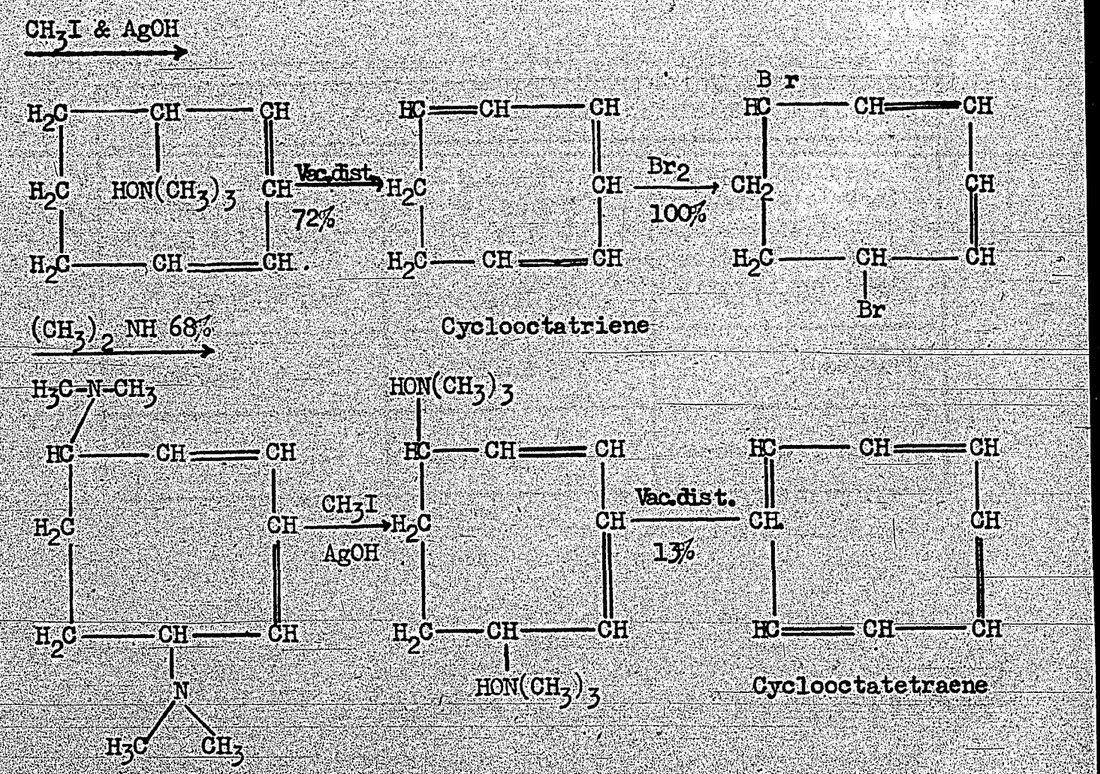
In this way, it has been possible to produce readily, directly from acetylene and in any desired quantity, Willstätter's Cyclooctatetraene which hitherto has been only a laboratory curiosity because of the extraordinary difficulty of obtaining it.

Cyclooctatetraene, the transformation products of which are also of commercial interest, will undoubtedly in future be produced technically in large quantities. The new synthesis offers, for the first time inter alia, a means of investigating the eight membered ring.

Willstätter and his collaborators 5) synthesized cyclooctatetraene for the first time by an extraordinarily assidious and difficult method, and for the purpose of checking Thiele's Valency Theory, investigated it most closely in respect of its unsaturated characteristics. In this case, they started from a natural product viz. pseudo-pelletierine, an alkaloid of the pomegranate series, in which the eight membered ring is already formed. By repeated exhaustive methylation double bonds were introduced by destilling the quaternary ammonium bases, yielding cyclooctadiene, which was converted to cyclooctatraene by bromination, exchange of bromine for dimethylamine, further exhaustive methylation and finally distillation of the free ammonium base. From 100 kg. pomegranate rind, 100 g. pseudo-pelletierine and, from this, 3-4 g. cyclooctatetraene was obtained.







B. Cyclooctatetraene.

1. Manufacture and physical properties.

We had already in 1940, made the observation that under pressure and by the action of certain nickel compounds, especially of nickel halogenides and nickel cyanide in the presence of solvents, tetrahydrofurane being most suitable, acetylene can be condensed to cyclopolyolefines, and particularly, to cyclooctatetraene. Very labile acetylene-nickel compounds first produced from the halogenides by the action of acetylene under pressure may be regarded as the effective catalysts. The formation of these nickel-acetylides may be facilitated, by the addition for example, of cyclic oxides, such as ethylene oxide, since the unstable nickel halogenalcoholates which are probably formed intermediately from the halogenides and ethylene oxide are readily split by the action of acetylene into the labile nickel-acetylides and ethylene halogen-hydrins.

This result is surprising from the point of view of thermodynamics, since cyclooctatetraene has a greater energy content than benzene.

Heat of combustion and formation		
	Heat of Combustion Q.P.	Heat of Formation (C=94.3 H=34.2)
Vinyl acetylene	569 Kcal/Mol	-57 Kcal/Mol
Benzene	783 Kcal/Mol	-12.4 Kcal/Mol
Cyclooctatetraene	1069 Kcal/Mol	-40 Kcal/Mol

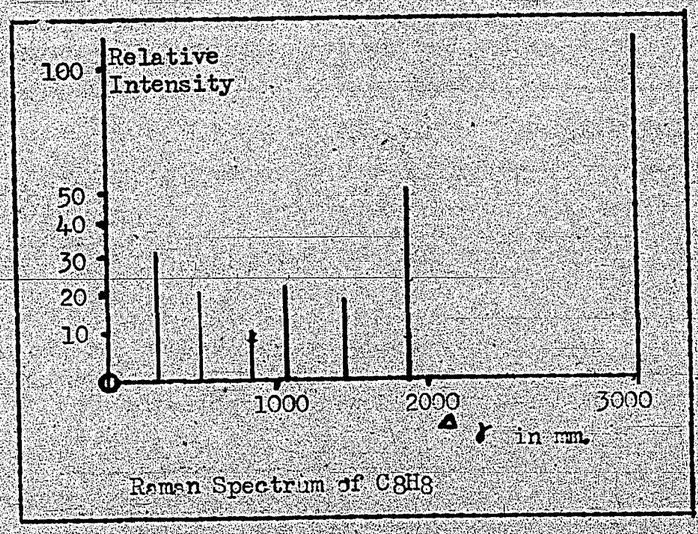
Heat of reaction on formation from acetylene			
	Q (liquid)	Q (gaseous form)	Q per Mol C_2H_2
2 C_2H_2 → Vinyl acetylene	+55.8 Kcal/Mol	+49.6 Kcal/Mol	+25 Kcal
3 C_2H_2 → Benzene	+153.8 Kcal/Mol	+164.4 Kcal/Mol	+49 Kcal
4 C_2H_2 → Cyclooctatetraene	+180.6 Kcal/Mol	+170.6 Kcal/Mol	+42.5 Kcal

Cyclooctatetraene is a golden-yellow liquid, with a boiling point of 142 - 143°C at 760 mm pressure, or 42.0 - 42.5°C. at 17 mm Hg and a melting point of -7.0°C.

The physical data found by Willstätter are somewhat different from those found by us, as is shown in the following table. The differences may be explained by the fact that we had available substantially larger quantities than Willstätter and it was therefore easier for us to determine the physical data with greater accuracy.

Physical data of Cyclooctatetraene	our data	Willstätters data	
bp. 760	142-143°	-	
bp. 17	42.0-42.5°	42.2-42.4°	
mp.	-7.0°	- 27°	
d^{0}_{4}	0.9382	0.943	
d^{20}_{4}	0.9206	0.925	
N^{20}_{D}	1.5290	1.5389	
Mol-Refr.	35.17	35.2	Calc. C_8H_8 = 35.08
Mol-Exalt.	-0.09	-0.12	
Dielectric constant ?.	2.74 20° 1.5×10^{6} Hg.	-	
Heat of combustion QP	1069.02 Kcal/Mol	-	
Dipolmoment. n.	$0.069.10^{-18}$ e.s.u.	-	

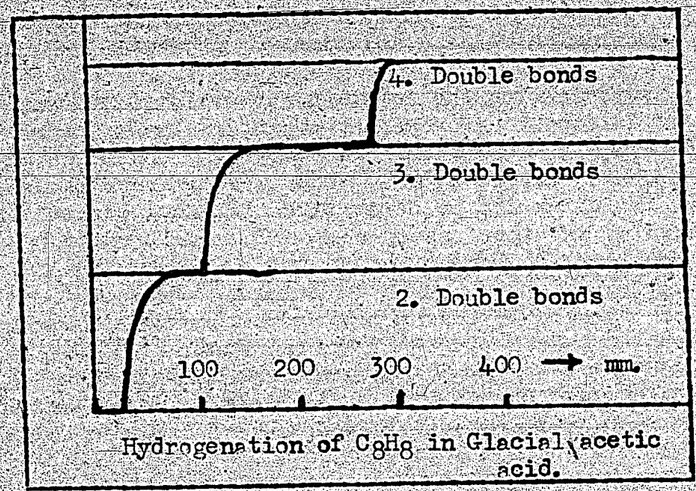
There is no doubt that in spite of the small differences, Willstätter's product and ours are identical and that both are 1.3.5.7- cyclooctatetraene. Beside the chemical behaviour to be described later, the dipole moment, which was found to be practically zero, and the Raman spectrum which exhibits 7 of the possible 42 Raman lines, indicate the symmetrical structure of the cyclooctatetraene-molecule.



Raman Spectrum of C_8H_8

The two strongest lines (Raman-frequencies 1673 and 3068 cm^{-1}) correspond, according to experience, to the "C-C" and "C-H" linkages. For benzene for comparison, the corresponding Raman-frequencies are 1605 and 3060 $cm.^{-1}$ respectively.

Furthermore, hydrogenation with Pd animal charcoal catalysts in glacial acetic acid demonstrates the required equality of the 4 double bonds of cyclooctatetraene.



Hydrogenation of C_8H_8 in Glacial acetic acid.

The last mentioned observations prove the high degree of symmetry of the golden-yellow hydrocarbon C_8H_8 and indicate strongly that the four double bonds present in the molecule are conjugated. The conception of Charles D. Hurd and Lewis R. Drake [6] postulating the presence of adjacent double bonds in Willstätter's cyclooctatetraene from their experiments with 1.2- and 2.3-dibrombutane and pyrogenic dissociation of 1.2- and 2.3 butanebis-(trimethylammonium)-hydroxides is therefor obsolete.

The olefinic character of cyclooctatetraene has already been demonstrated by Willstätter. According to Erich Huckel's [7] recent discoveries, the electron shell of cyclooctatetraene comprises 8 π - electrons compared with the 6 π - electrons of benzene which represent a closed shell. The olefine nature of cyclooctatetraene [8] is explained in this way and by the arrangement of the carbon atoms of the eight membered ring, which presumably does not lie in a plane. In the infra red, visible and ultra violet spectral regions, cyclooctatetraene shows the following absorption:-

Diagram missing from original

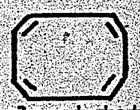
2. Reactions of Cyclooctatetraene.

Cyclooctatetraene, because of its olefine character, is very labile towards various reagents. As already observed by Willstätter, it precipitates yellow flakes when exposed to air. It is very easily attacked by oxidizing agents. Halogens add rapidly. It is possible to subject it to diene synthesis when characteristic adducts are formed. It is transformed into a mixture of dimerides and resin like substances on standing for a sufficiently long time, which may be shortened by heating. Crystalline addition compounds are formed by the action of aqueous silver nitrate-solution and of ammoniacal copper-chloride - ammonium chloride - solution.

Whilst investigating the reactions of cyclooctatetraene it was found that it is capable of reacting structurally differently towards different reagents. Whilst in some reactions, the eight membered ring remains unaltered, in others, aromatization results exceedingly readily with the formation of derivatives of ethyl benzene and p-Xylene; again, in other cases, compounds result which have both a six and a four-membered ring.

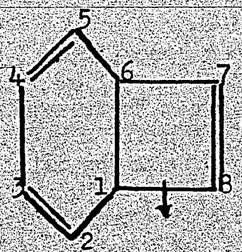
Cyclooctatetraene is thus able to react in the following ways: (1) as an eight membered ring; (2) as a bicyclo-(0.2.4)-octatriene (2.4.7); and (3) as 1.2.4.5 - dimethylenecyclohexadiene-2.5. In case (2) splitting of the four-membered ring may occur with the formation of derivatives of ethylbenzene.

Ring systems to which Cyclooctatetraenes may give rise during reaction.



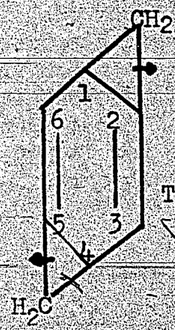
Type I

cyclooctatetraene



Type II

Bicyclo-(0.2.4)-octa-triene-(2,4,7)



Type III

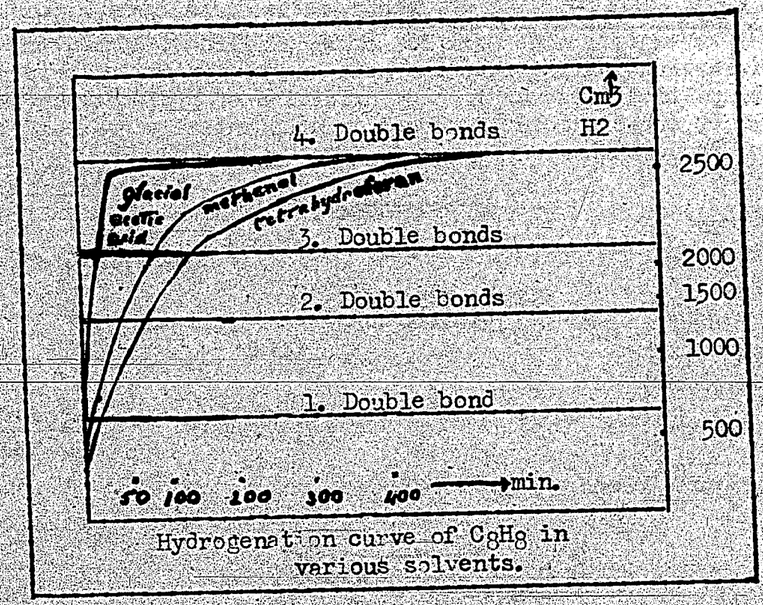
1,2-4,5- Dimethylencyclohexadiene-2,5.

I. Reactions of cyclooctatetraene of type I.

(a) Catalytic hydrogenation.

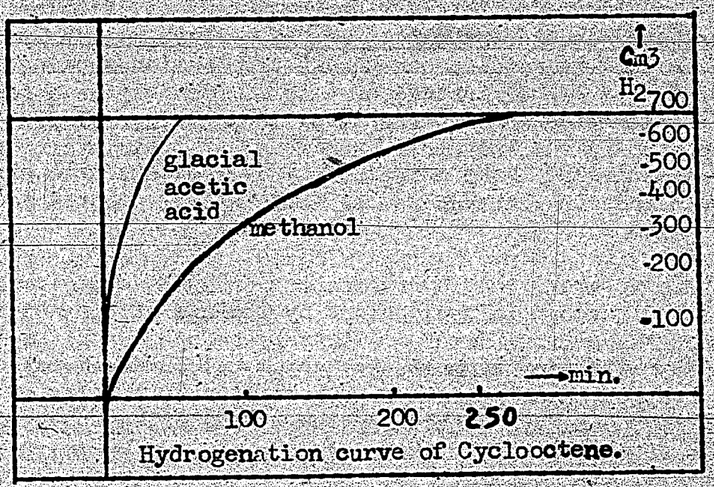
As already discovered by Willstätter, cyclooctatetraene is transformed into cyclooctane on catalytic hydrogenation using a Pt-catalyst. The eight membered ring is, thus, preserved in this case. Hydrogenation occurs readily not only with precious metal catalysts but also with other catalysts, such as nickel, under pressure. We also made the same observation as Willstätter, namely, that the resulting cyclooctane has a lower melting point than that given in the literature, our purest preparation showing a melting point of + 11.8°C instead of + 13°C, probably as a result of a slight aromatization through reactions of type II or III. (Occurrence of p-xylene and ethyl benzene or of their hydrogenation products respectively). By oxidation of cyclooctane with nitric acid, we obtained, in the same way as Willstätter, suberic acid in 40% yield.

It became apparent that hydrogenation of cyclooctatetraene can only be carried through to cyclooctane by the use of non-acidic solvents such as methanol, ethanol, tetrahydrofurane and the like, and also partially with, for example, palladium-calcium-carbonate as catalyst. Under these conditions, absorption of 3 moles of hydrogen is effected very rapidly, whilst for the 4th mole the absorption is ten times slower.



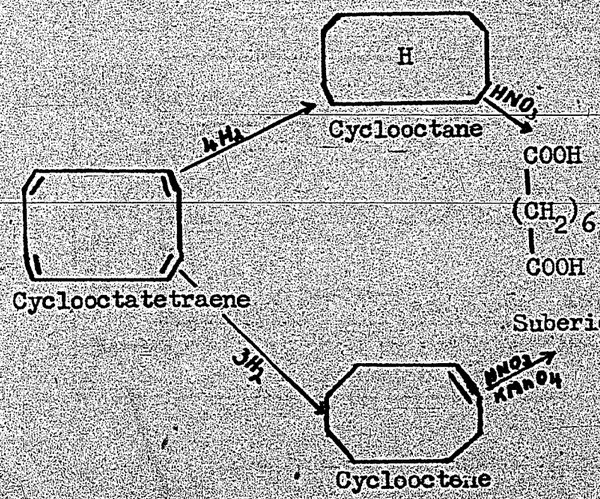
Hydrogenation curve of C_8H_8 in various solvents.

If the hydrogenation is stopped after absorption of 3 moles of hydrogen, cyclooctene having a boiling point of 140°C can readily be isolated in approximately 90% yield. Comparative hydrogenation tests using the cyclooctene so obtained, in alcoholic and acetic solution resulted in an absorption of hydrogen which was ten times faster in the latter case than in alcoholic solution, confirming the effect also observed in other tests, of the solvent on catalytic hydrogenation.

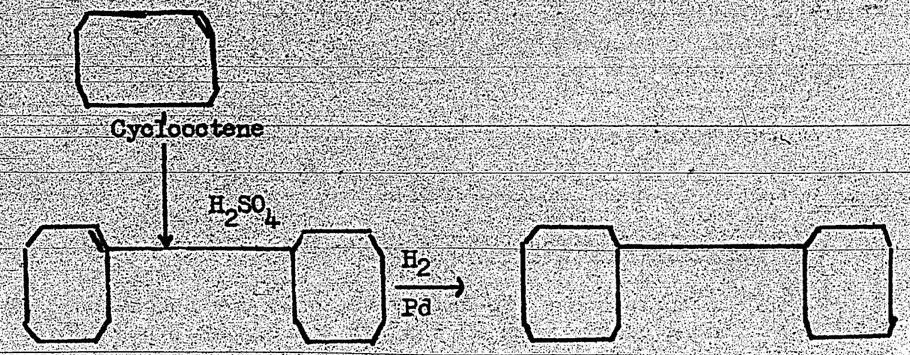


Hydrogenation curve of Cyclooctene.

Cyclooctene can easily be converted, by means of bromine, into the well known dibromide $C_8H_{14}Br_2$ of Bp 123-124°C. 15 mm. and by means of Perbenzoicacid, into the well known crystalline oxide $C_8H_{14}O$ of melting point 45°C. By oxidation of cyclooctene with nitric acid, suberic acid is formed in substantially greater yield than from cyclooctane.



Being an olefine, it can be subjected to the known olefine reactions and thus represents a convenient starting material for substituted cyclooctenes and for cyclooctanone. By the action of dilute acids, such as sulphuric acid, it is condensed to cyclooctenyl-cyclooctane, which can be converted by hydrogenation into dicyclooctane, already described by Ruzica [9].

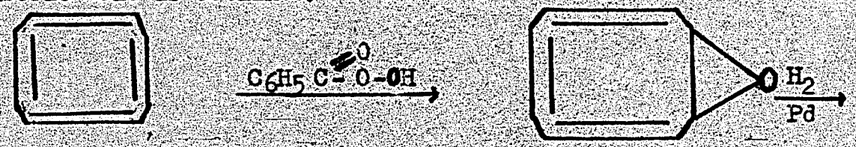


Cyclooctenyl-cyclooctane

Dicyclooctane

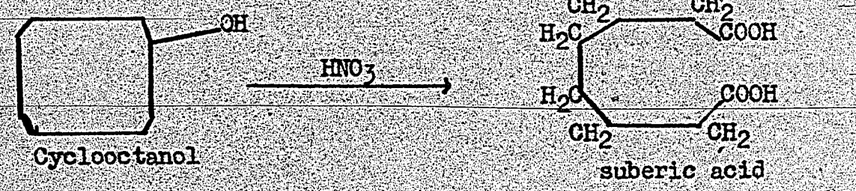
(b) Reactions with peracids.

By treatment of cyclooctatetraene with one or several moles of perbenzoic acid in chloroform solution (Prileschajew) a single oxygen atom is introduced, the almost colourless oxide of the composition C_8H_8O being formed which absorbs 4 moles of H_2 to yield cyclooctanol, when catalytically hydrogenated. By oxidation with nitric acid suberic acid is obtained.



Cyclooctatetraene

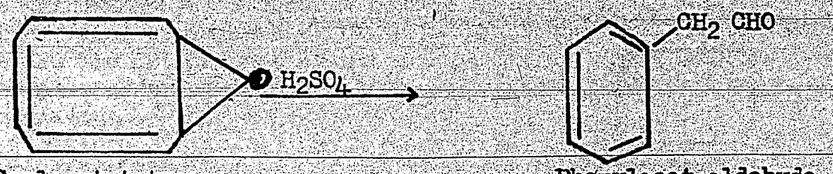
Cyclooctatetraene-oxide



Cyclooctanol

suberic acid

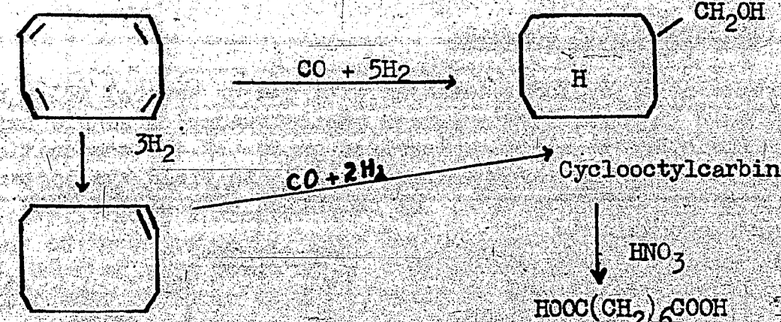
If cyclooctatetraene oxide is heated with a few drops of dilute sulphuric acid, phenylacetaldehyde is formed quantitatively in a violent reaction (boiling) involving aromatization.



Cyclooctatetraene — Phenylacet-aldehyde

(c) Reaction with ($CO + H_2$).

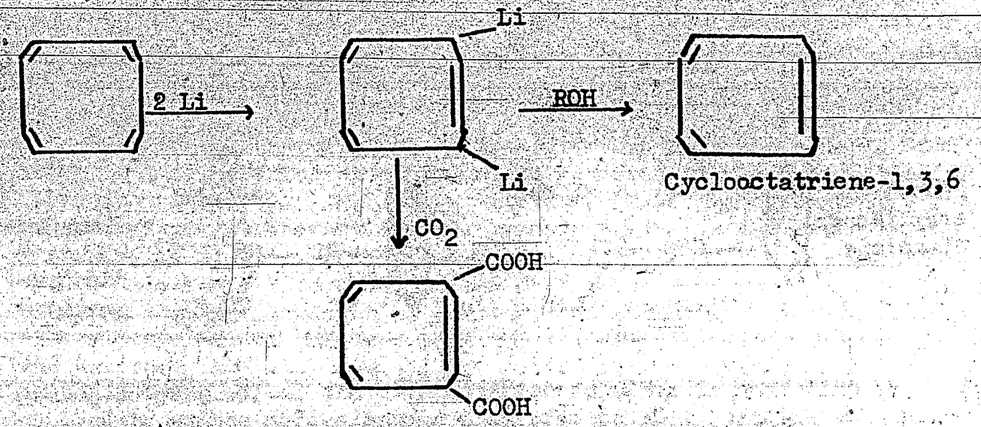
Under the influence of Co-catalysts, only one double bond of the cyclooctatetraene reacts with $CO + H_2$ in the manner of the "Oxo-reaction" forming the carbinol, the other three double-bonds being saturated. The following diagram shows the reaction sequence which leads to cyclooctylcarbinol of Bp = 106°C/7 mm.



Cyclooctylcarbinol can also be obtained by the "Oxo-reaction" from cyclooctene and water gas ($CO + H_2$).

(d) Effect of alkali metals.

Cyclooctatetraene combines with two atoms of an alkali metal, whilst preserving the eight membered ring. By the action of CO_2, a crystalline yellow dicarbocylic acid is formed, which sublimes at 220°C at 1.5 mm Hg pressure. With alcohols, the alkali metal atoms are replaced by hydrogen with the formation of Willstätter's cyclooctatriene. The experiments were carried out with metallic lithium.



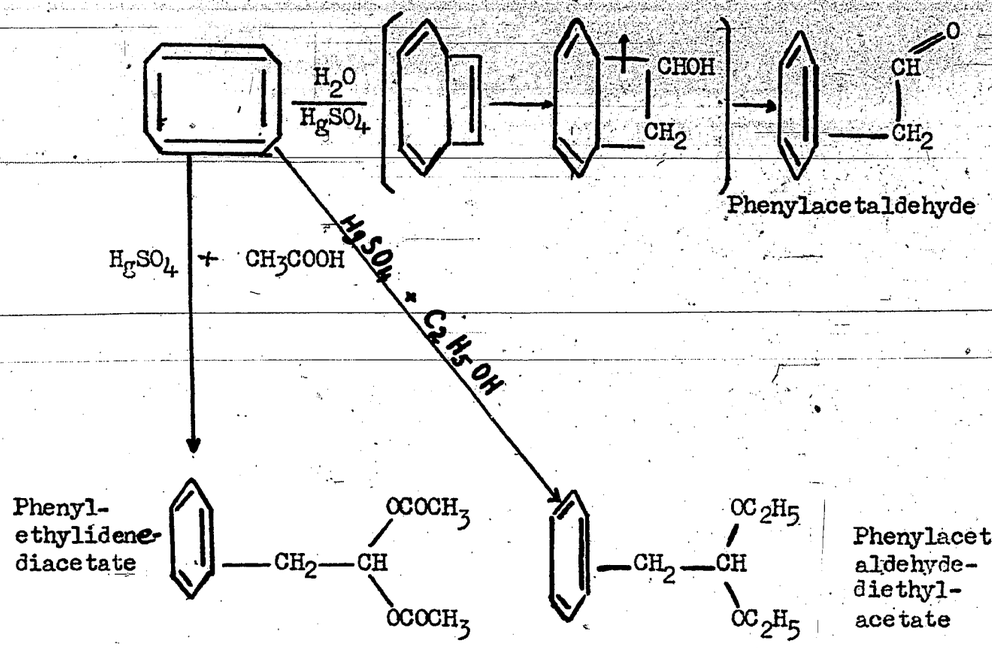
Cyclooctatriene-1,3,6

II. Reactions of cyclooctatetraene of types II and III.

(a) Aromatization of type II in which the four membered is split, and of type III.

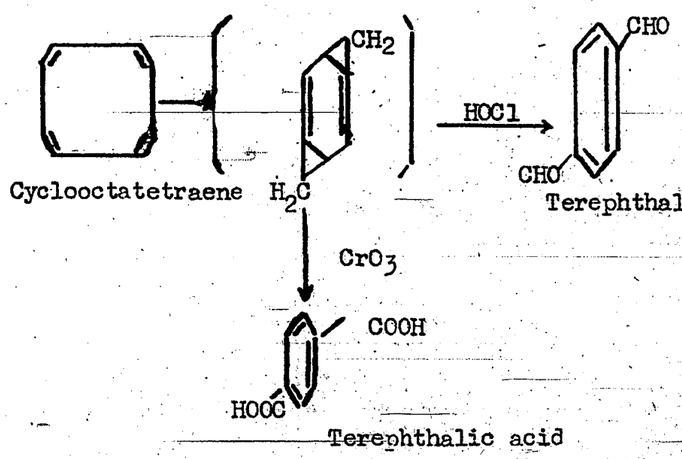
On treatment of cyclooctatetraene with reagents having an oxidizing effect, aromatization of type II in which the four membered ring is split, or of type III always occurs.

If cyclooctatetraene in aqueous suspension or emulsion is treated with mercury salt solutions, for example, $HgSO_4$, a white precipitate is immediately formed, which blackens through separation of metallic mercury, forming a colourless oil C_8H_8O which has a boiling point of 221°C. The oil is identical with phenylacetaldehyde. The yield is practically quantitative. If in this reaction the water is replaced by glacial acetic acid or alcohol, the well known phenylethylidendiacetate or diethylacetal of phenylacetaldehyde respectively is immediately formed.

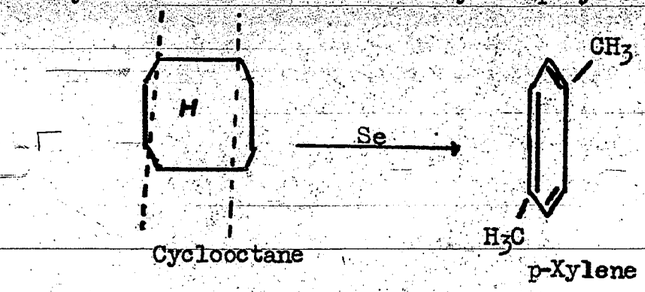


By oxidation of cyclooctatetraene with air in the gaseous phase using, for example, V_2O_5 as catalyst, aromatization occurs with formation of benzoic acid in almost quantitative yield. Benzaldehyde as well as benzoic acid is formed with permanganate in aqueous solution.

With hypochlorous acid in alkaline solution as oxidizing agent, aromatization of type III takes place with formation of derivates of p-xylene. Terephthaldialdehyde, benzaldehyde and benzoic acid are formed. Chromic acid in glacial acetic acid produces terephthalic acid and some benzaldehyde.



A similar aromatization of the eight membered ring to the benzene ring has already been observed by Ruzicka [10] in the dehydrogenation of cyclooctane with selenium to yield p-xylene.



(b) Reactions of type II preserving the four membered ring.

Many of the reactions of cyclooctatetrene examined proceed with structural transformation of the eight membered ring and formation of derivates of bicyclo - (0.2.4)-octatriene -(2.4.7) according to reaction type II, whereby the four membered ring is preserved. In this mode of reaction are included, primarily, the transformations of cyclooctatetraene with halogens and dienophile reagents. By carefully studying the reactions of the first formed adducts, and their decomposition products, it has been possible to determine their composition, and to prove that they can all be derived from bicyclo-(0.2.4)-octane as basic hydrocarbon.

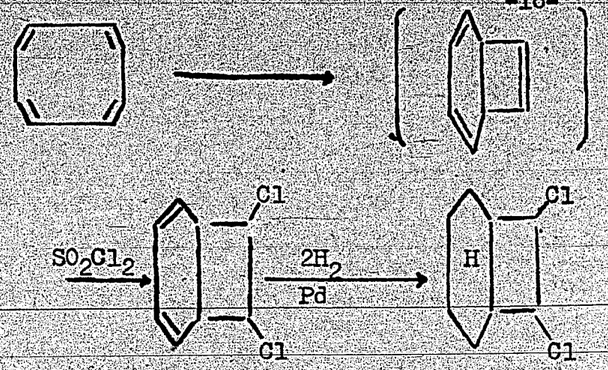
α Halogenation of cyclooctatetraene.

The halogenation products, so far prepared, can be seen from the following table:-

Halogenation products of Cyclooctatetraene		
$C_8H_8Br_2$	Br_2 in $CHCl_3$ at -20°C (Willstätter)	mp. 71°C
$C_8H_8Br_2$	Br_2 in $CHCl_3$ at 0° - 5°C.	bp. 1 mm / 90°C.
$C_8H_8Br_4$ $C_8H_8Br_4$	$2Br_2$ in $CHCl_3$	mp. 94°C. mp. 147 - 148°C.
$C_8H_8Br_6$	$3Br_2$ in $CHCl_3$	mp. 156°C. + liquid isomer.
$C_8H_8Cl_2$	SO_2Cl_2 in $CHCl_3$	bp. 0.5 mm / 62°C.
$C_8H_8Cl_4$ $C_8H_8Cl_4$	2 SO_2Cl_2 in $CHCl_3$ or CH_2Cl_2	mp. 111° - 112°C. bp. 1 mm / 126°-128°C.
$C_8H_8Cl_6$	Cl_2 in $CHCl_3$ at 0° - 5° C.	mp. 126° - 127°C. + liquid isomer.

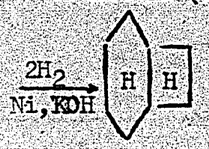
Willstätter has already described a crystallized dibromide of cyclo-octatetraene which has a melting point of 71°C. and which he obtained by brominating cyclooctatetraene in chloroform at temperatures of approximately -20°C. We were able to confirm this result, but found that this dibromide is very unstable, readily resinifies and rapidly disintegrates with evolution of hydrogen bromide and rise in temperature. By varying the conditions of bromination (temperature) we were, however, able to obtain a liquid dibromide $C_8H_8Br_2$ of bp. = 90°C./1 mm. which can be distilled in vacuum. By the use of 2 moles or 3 moles respectively of bromine, amongst others, two tetrabromides $C_8H_8Br_4$ of m.p. 94°C. and 147-148°C. respectively were obtained which crystallise to form colourless prisms and a hexabromide $C_8H_8Br_6$ of m.p. 156°C. which is fully saturated and represents the highest degree of bromination, which also crystallized to form colourless prisms. An octabromide compound never occurred.

Chlorination of cyclooctatetraene similarly led to well defined products. With one mole of sulphuryl chloride, a fluid dichloride $C_8H_8Cl_2$ of bp. 62°/0.5 mm. is produced, and with two moles, a solid tetra-chloride $C_8H_8Cl_4$ of mp. 111-112°C. as well as a fluid tetra-chloride of bp. 126-128°C./1 mm. Complete chlorination is in this case also represented by a saturated hexachloride $C_8H_8Cl_6$ mp. 126-127°C which can best be obtained by introduction of chlorine into a chloroform solution of cyclooctatetraene at 0-5°C. Mixed halogen derivates may also be obtained. Iodine derivates, however, have so far not been obtainable. The result, that the fully halogenated derivative, the hexa-halogen compound, is saturated and that the dihalogen-compound absorbs only two moles instead of three moles of hydrogen as expected on catalytic hydrogenation yielding a saturated dihalogenide, leads to the assumption, that structural change of cyclooctatetraene has occurred during halogenation, and that the saturated hydrocarbon C_8H_{14} which forms the basis of the halogenation products must be of a bicyclic nature. A bicyclic system has, thus, been formed from the eight membered ring of cyclooctatetraene due to ring contraction, the bicyclic system containing a six and a four membered ring, as will be proved in the following:-



7.8-Dichlor-Bicyclo-(0.2.4)-octadiene-(2.4)

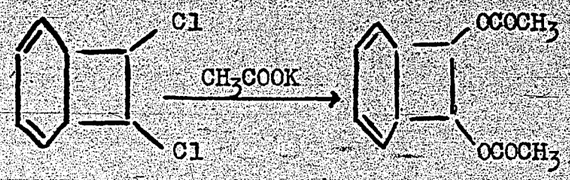
7.8-Dichlor-bicyclo-(0.2.4)-octane



Bicyclo-(0.2.4)-octane

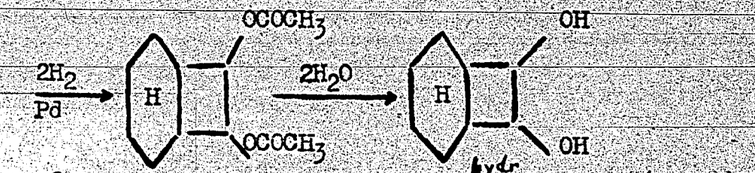
By hydrogenation of the dichloride $C_8H_{12}Cl_2$ in an alkaline medium under pressure, the basic hydrocarbon C_8H_{14}, namely bicyclo -(0.2.4)-octane of bp. 136°C. may be obtained in a pure state..

The proof of its constitution, as well as the reason for the position of the halogen atom, as indicated in the above formula, was obtained as follows. If the dichloride $C_8H_{14}Cl_2$ is allowed to react with potassium acetate in glacial acetic acid, a crystalline diacetate $C_8H_8(OCOCH_3)_2$ of mp. 66°C. is obtained, which absorbs 2 moles of hydrogen during hydrogenation and is thereby changed into the saturated diacetate $C_8H_{12}(OCOCH_3)_2$ (bp. 105°/0.4 mm.) which by subsequent saponification with methyl alcoholic hydrochloric acid quantitatively produces a glycol $C_8H_{14}O_2$ of mp. 142°, crystallising as colourless flakes. The latter oxidises to cis-hexahydrophthalic acid from which the orthofusion of the six-and four-membered rings as well as the position of the two Cl-atoms in the four membered ring becomes evident. This is also indicated by the formation of cis-hexa-hydrophthalaldehyde through fission of the glycol $C_8H_{14}O_2$ by oxidation with lead-tetra-acetate according to Criegee:



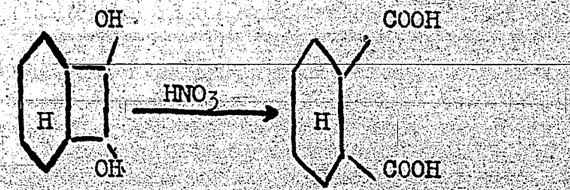
7.8-dichlor-Bicyclo-(0.2.4)-octadiene-(2.4)

7.8-diacetoxy-Bicyclo-(0.2.4)-octadiene-(2.4)



7.8-Diacetoxy-bicyclo-(0.2.4)-octane

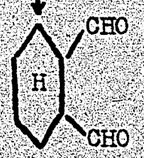
7.8-Dihydroxy-bicyclo-(0.2.4)-octane



7.8-Dihydroxy-Bicylo-(0.2.4)-octane

Cis-Hexahydro-phthalic acid

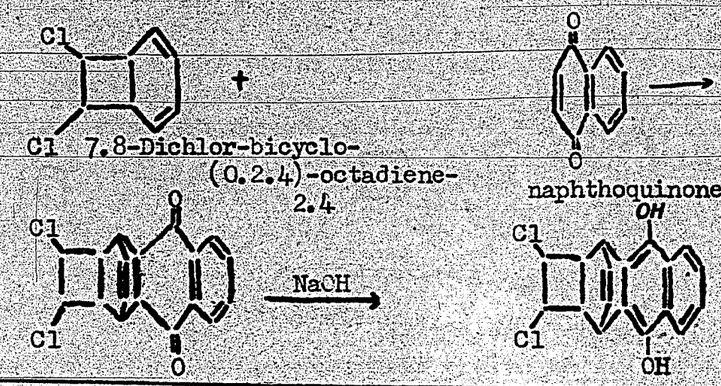
$Pb(OOC-CH_3)_4$



Cis-Hexahydro-phthalic aldehyde

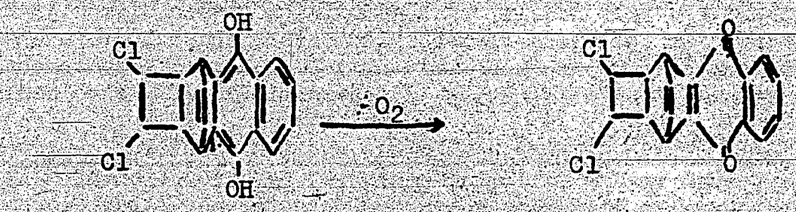
Proof of the presence of the four membered ring can also be furnished by other methods. If the dichloride $C_8H_8Cl_2$ which also contains two conjugated double bends in the six-membered ring and thus can undergo diene addition is caused to react with naphthoquinone, an adduct of mp. 221°C. is formed which crystallises as colourless needles. Its solution in alkali is red and is readily oxidised in air to the anthraquinone, the anthraquinone crystallizing as a light yellow substance which melts at 170-180°C. with decomposition and loss of a volatile substance, resolidifying upon further heating to approximately 220°C. and finally remelting at 275-280°C.

The decomposition products consist partly of anthraquinone and partly of the so far unknown 1.2 dichlorcyclobutene -3 of bp. 133°C. and mp. 44°. The complete sequence of the reaction is reproduced in the following diagram:



7.8-Dichlor-bicyclo-(0.2.4)-octadiene-2.4

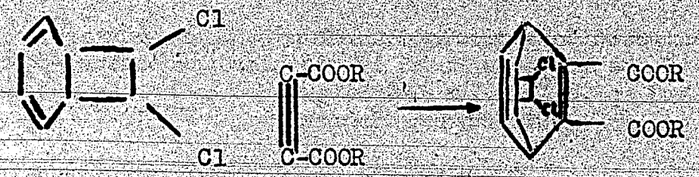
naphthoquinone



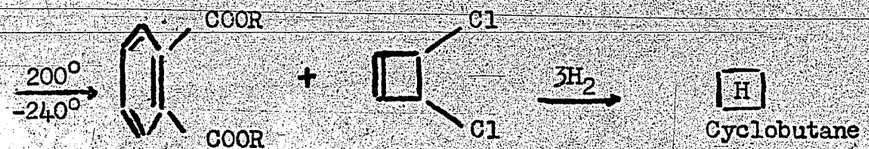
anthraquinone

1.2-Dichlor-cyclobutene-3

The same behaviour on thermal decomposition is shown by the adduct of the dichloride compound of cyclooctatetraene with acetylene-dicarboxylic-ester furnishing, quantitatively, diethyl-phthalate and 1.2- dichlorbutene - 3:-



Acetylene dicarboxylic ester



Phthalic ester

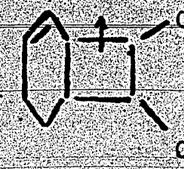
1.2-Dichlor-cyclobutene-3

Cyclobutane

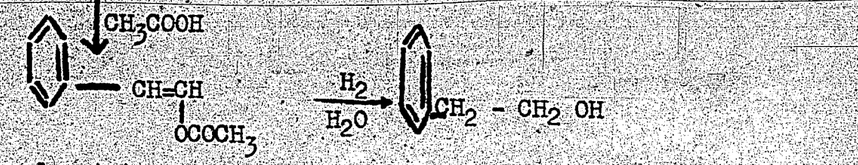
These results agree with observations of Diels [11] and with the Alder-Rickert [12] rule namely, that adducts of cyclic dienes with naphthoquinone or acetylene-dicarbocylic ester, which contain an endo- ethylene bridge, are thermally unstable and can be decomposed into an aromatic with elimination of the remainder of the molecule.

That the cyclic dichloride $C_4H_4Cl_2$ has the constitution given above was proved in the following manner:- By absorption of only one mole of bromine it can be converted into the saturated dibromide $C_4H_4Br_2Cl_2$ bp. = 119-120°C./18 mm. It, thus, contains only one double bond and must, therefore, be cyclic. By oxidation with nitric acid, fumaric acid is formed, the two C-atoms carrying the Cl-atoms being oxidized to carboxyl groups. By catalytic hydrogenation in the presence of alkali, 3 moles H_2 are absorbed with formation of cyclobutane. The presence of a four-membered ring is thereby proved beyond doubt.

The behaviour of the dihalogen compound of cyclooctatetraene towards glacial acetic acid, alcohols and alcoholates is noteworthy. Whilst aromatization occurs in the first two cases, a surprising structural transformation takes place in the latter. If the dichloride compound $C_8H_8Cl_2$ is heated with glacial acetic acid, a fair amount of styryl acetate is produced with formation of hydrochloric acid and by hydrogenation and saponification the styryl acetate is converted into phenyl-ethyl-alcohol.



7.8-Dichlor-bicyclo-(0.2.4)-octadiene-(2.4)



Styrylacetate

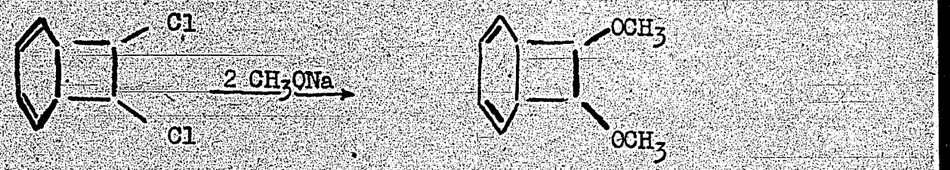
β-Phenyl-ethyl alcohol

Similarly by treatment with alcohols with heating, the dimethyl-ether of the phenylglycol is produced with aromatization and fission of the four-membered ring. (Identified by comparison with the dimethylether obtained by methylation of phenylglycol).

7.8-Dichlor-bicyclo-(0.2.4)-octadiene-(2.4)

Phenylglycol-dimethylether

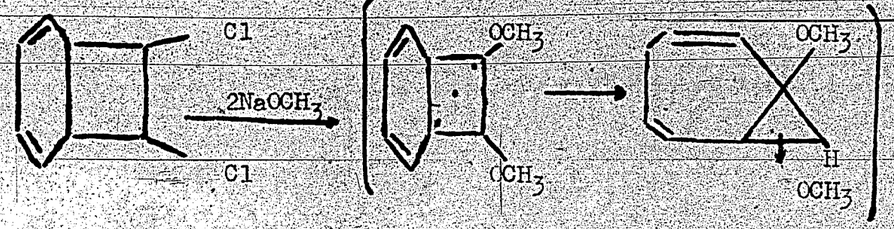
Also in the treatment of the dichloride $C_8H_8Cl_2$ with alkaline-alcoholates simple exchange of the two halogen atoms for alkoxy groups does not take place, as suggested below,

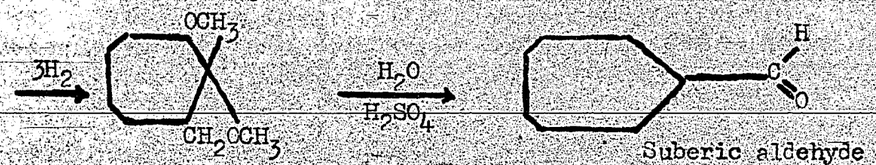


7.8-Dichlor-bicyclo-(0.2.4)-octadiene-(2.4)

7.8-Dimethoxy-bicyclo-(0.2.4)-octadiene-(2.4)

because the dialkyl ether formed absorbs 3 moles of H_2 on catalytic hydrogenation instead of the expected 2 moles and does not undergo diene addition as would be expected of a substance of the constitution at first assumed. Close study of the hydrogenation showed that a surprising structural transformation occurs during the reaction of the dichloride $C_8H_8Cl_2$ with alcoholates, leading finally to suberic aldehyde as can be seen from the following scheme:-



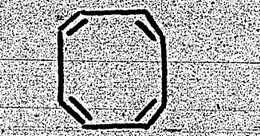


The suberic aldehyde was converted to the semi-carbazone of mp. 194°C, or after oxidation, into suberic amide mp. 194°C. being identified in both cases, by admixture with the corresponding synthetically prepared substances.

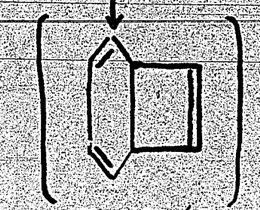
β Diensynthesis with cyclooctatetraene.

The four conjugated double bonds of cyclooctatetraene might be expected to react with 2 moles of a dienophilic reagent. This, however, is not the case. In all the reactions we have studied, (addition of maleic anhydride, acrylic acid, quinone, naphthoquinone and the like) cyclooctatetraene reacts with the dienophilic reagent only in the ratio 1:1. Also, the adducts thus obtained do not enter into further diensynthesis. The temperature of reaction is generally rather high: it is usually necessary to heat up to 100°C. or to force the reaction in the autoclave at a higher temperature. All the diene adducts of cyclooctatetraene show the common feature that they absorb, during hydrogenation, one mole of H_2 less than would be expected of the product of a simple addition. A secondary reaction must therefore have occurred during the diensynthesis, with structural transformation of the eight-membered ring of cyclooctatetraene, in the course of which one double bond disappears. We studied this problem more closely in connection with the maleic anhydride adduct of mp. 166°C. which is formed quantitatively from cyclooctatetraene and maleic anhydride. We found that diene addition, similar to halogenation,

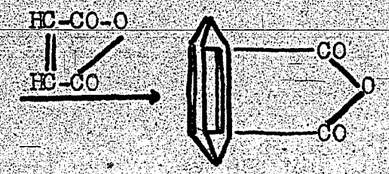
passes through the hypothetical intermediary stage of the bicyclic (0.2.4)-octatriene-(2.4.7).



Cyclooctatetraene



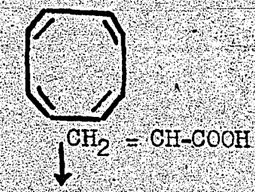
Bicyclo-(0.2.4)-octatriene-(2.4.7)

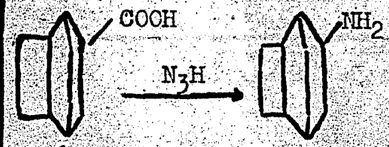


3.6-Endocyclobutadienylene cyclohexen-4-dicarboxylic acid-1.2-anhydride.

The two double bonds of the final product differ in respect of their speed of hydrogenation, so that partial hydrogenation is possible.

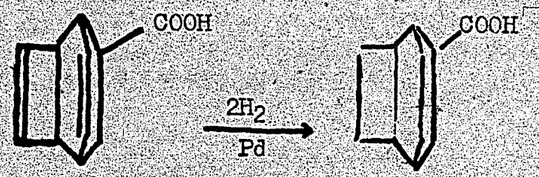
For further proof of the constitution, the acrylic acid adduct, which melts at 113°C, was prepared, hydrogenated and decomposed through the azide and amine to a saturated tricyclic hydrocarbon $C_{10}H_{16}$.





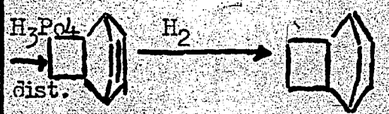
3.6-Endocyclobutylenecyclohexane-carboxylic acid-1.

3.6-Endocyclobutylenecyclohexylamine-1



3.6-Endocyclobutadienylene-cyclohexene-4-carboxylic acid-1

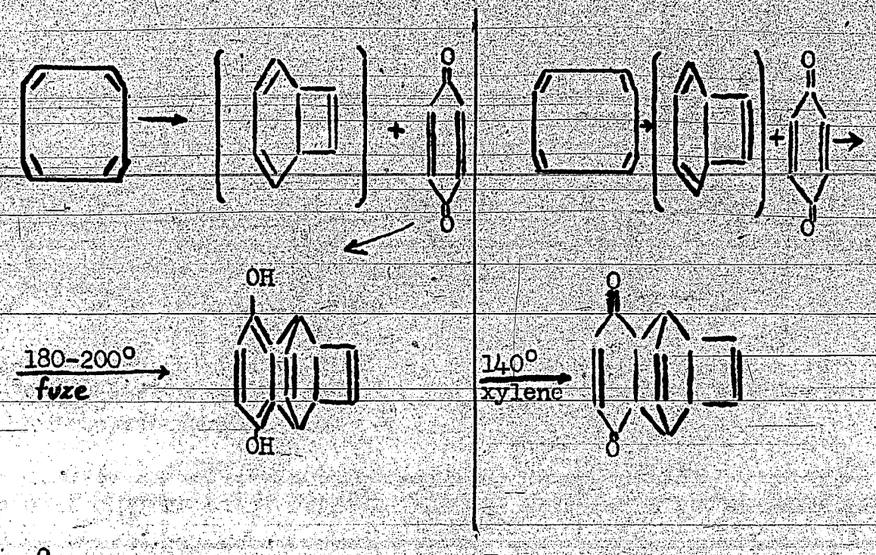
3.6-Endocyclobutylen-cyclohexane-carboxylic acid-1.

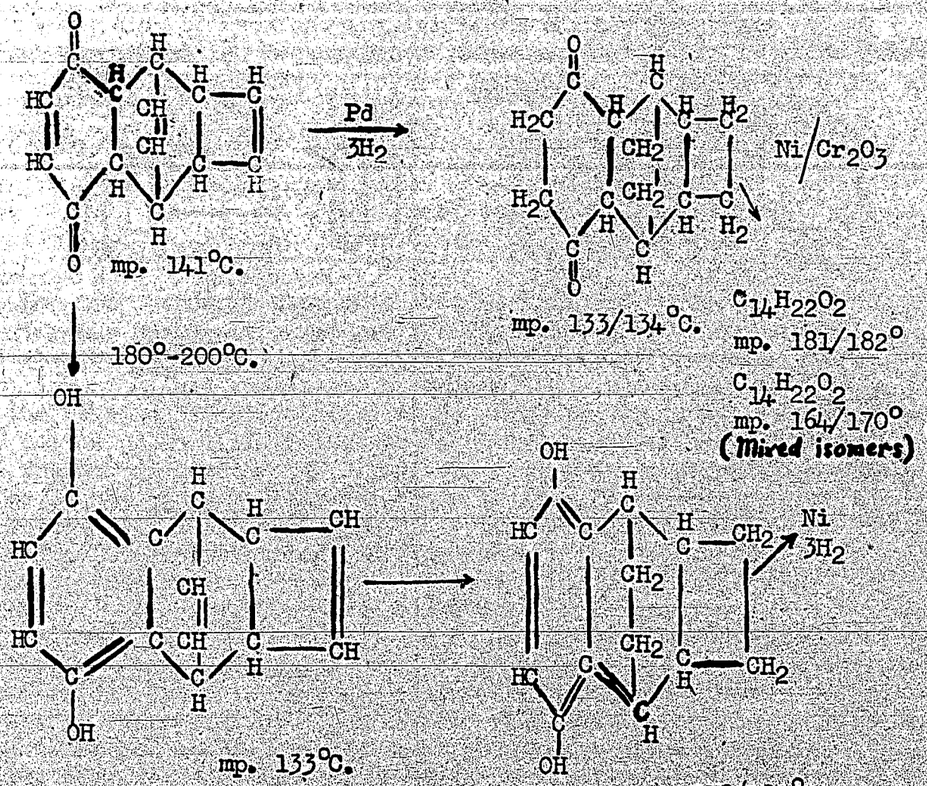


3.6-Endocyclobutylene-cyclohexene-1

3.6-Endocyclobutylenecyclohexane (2.5 Endo ethylene-Bicyclo-(0.2.4)-octane.

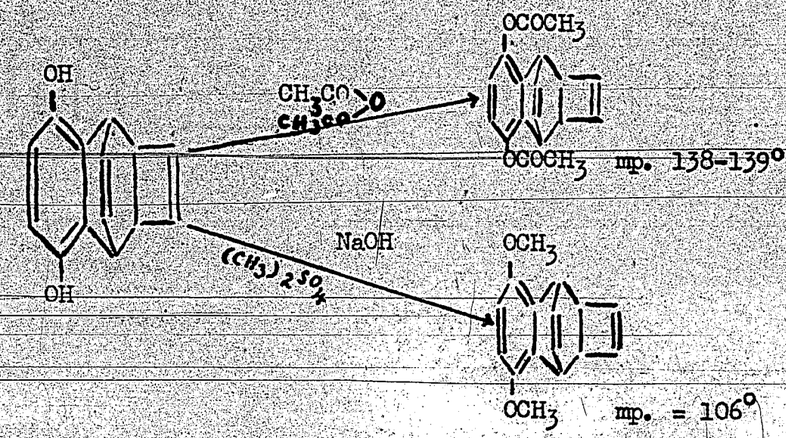
Quinone reacts quite analogously with cyclooctatetraene. Thereby, depending on the conditions of reaction, either the quinonoid or benzenoid form is produced.



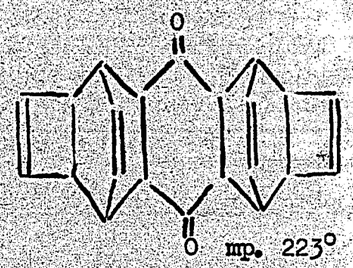


$C_{14}H_{22}O_2$
mp. 181/182°
$C_{14}H_{22}O_2$
mp. 164/170°
(Mixed isomers)

For further characterization of the new compounds, we prepared also the acetate and methyl ether:



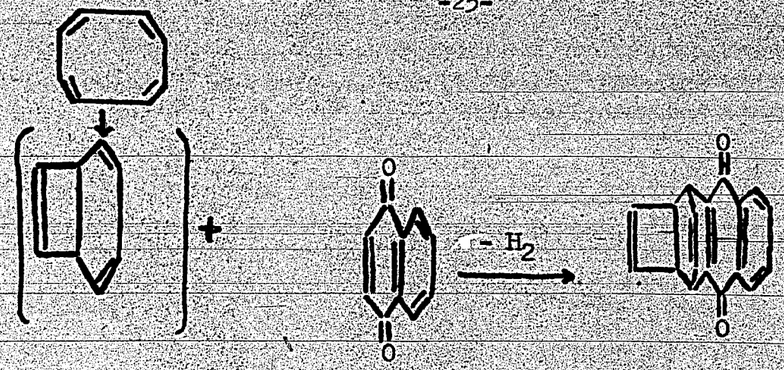
Benzoquinone also adds, to a lesser degree, to two moles of cyclooctatetraene, whereby the following product is formed:



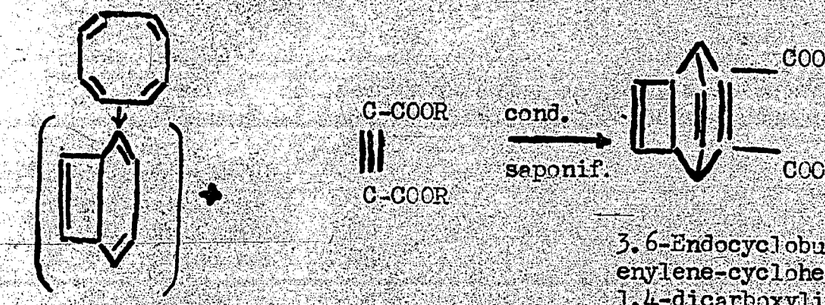
addition product from 2 mol/cyclooctatetraene and 1 mol quinone.

mp. 223°

Furthermore, adducts were formed with naphthaquinone and acetylene-dicarboxylic ester, which disintegrate into anthraquinone and phthalic acid respectively by thermal decomposition. Instead of the unknown cyclobutadiene also to be expected, only resinous polymerization products were obtained.



1.4-Endocyclobutadienylene-1.4-dihydroanthraquinone



3.6-Endocyclobutadienylene-cyclohexadiene-1.4-dicarboxylic acid-1.2.

3. Polymerization of cyclooctatetraene and its dihalogenation products.

Cyclooctatetraene and its dihalogenation products change into polymeric products when left standing for a comparatively long period which, however, may be shortened by heating. Only the dimeric polymerization products have previously been carefully examined. Dimeric polymerides are easily obtainable by boiling cyclooctatetraene for several hours under reflux. Thereby, a mixture of at least two structurally isomeric hydrocarbons of the formula $C_{16}H_{16}$ is obtained of which one melts at 43°C., the other being a viscous oil which boils at 297°C. (Separated by addition of ether to the mixture of hydrocarbons, and cooling). The solid hydrocarbon, towards catalytic hydrogenation, contains three double bonds, (the isomeric liquid hydrocarbon only two double bonds). However, by carrying out the dimerisation in absence of air (nitrogen) only the liquid hydrocarbon $C_{16}H_{14}$ is formed. By hydrogenation, a liquid hydrocarbon $C_{16}H_{22}$ of bp. 140°C. at 1 mm. is formed from the solid hydrocarbon $C_{16}H_{16}$, whilst the hydrogenation of the liquid hydrocarbon provides a solid hydrocarnon $C_{16}H_{20}$ of mp. 30-35°C.

The dimerization of cyclooctatetraene undoubtedly represents a diensynthesis. It is certain that in this case aromatization does not occur, since no aromatic decomposition products, such as benzoic acid, phthalic acid and the like were observed. No results are available concerning the constitution of the dimers.

It may be supposed that the unsaturated hydrocarbon is formed by dienecondensation of 2 moles of cyclooctatetraene in the following manner:

2 Mols. Cyclooctatetraene as Bicyclo-(0.2.4.)-octatriene-(2.4.7). $C_{16}H_{16}$ (liquid)

The reactivity of the dimeric cyclooctatetraene with CO and H_2O under the catalytic action of nickel carbonyl is worth noting. From the liquid modification, a dicarboxylic acid is formed of which the dimethyl ester boils at 200 - 205°C. at 0.6 mm. Hg. The solid modification, containing 3 double bonds, by the same treatment, also yield a dicarboxylic acid, whose dimethyl ester has approximately the same boiling point. If cyclooctatetraene is itself subjected to the same reaction, the same dicarboxylic acids are produced, probably owing to the dimerization of the cyclooctatetraene.

The liquid dihalogenation products change analogously into readily crystallizing dimerides $C_{16}H_{16}Cl_4$ of mp. 190°C. and $C_{16}H_{16}Br_4$ of mp. 218°C.

The work on the configuration of the various diene adducts of cyclooctatetraene mentioned has begun and has already led to noteworthy results, but has had to be interrupted for the time being.

III. Possibilities of application of cyclooctatetraene.

Based on the preliminary work, so far accomplished, it may be already predicted that cyclooctatetraene will become of considerable technical importance. The diene adducts are, for example, excellent starting materials for valuable raw materials for varnishes. The suberic acid obtainable from cyclooctatetraene through cyclooctane and cyclooctene respectively, is of great interest in the field of polyamides. Above all, cyclooctene itself, the double bonds of which are open to all the reactions of ethylene chemistry, points a new route into the chemistry of the eight-membered ring and its transformation products. It is possible, for instance, to synthesize caproic lactam from cyclooctene → cyclooctanol → cyclooctanone → cyclooctanone-oxime, a very desirable cornerstone in the chemistry of polyamides.

Furthermore, it may be expected that the new synthesis of cyclooctatetraene which is now easily obtainable will provide a new starting material for alkaloid synthesis.

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FINAL REPORT No. 392.
ITEM No. 21.

WELDING OF ALUMINIUM AND ALUMINIUM ALLOYS with particular reference to the MANUFACTURE OF PRESSURE VESSELS.

Brooks, H. "1

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WELDING OF ALUMINIUM AND ALUMINIUM ALLOYS WITH PARTICULAR REFERENCE TO THE MANUFACTURE OF PRESSURE VESSELS

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(1946)

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE,
32, Bryanston Square, W.1.

34 p. diags

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1. REPORTS ON VISITS TO VESSEL FABRICATORS

1.1. Wilhelm Schmidding, Köln-Niel

Schmiddings took over their spacious factory buildings, which were previously the town salvage department, in 1940. They have recently fused with Imbert G.m.b.H., and the new Company is called Amagen Imbert-Schmidding. They intend, in future, to make gas-producers for cars and small vessels, in addition to the usual work carried out by Schmiddings.

During the war, Schmiddings have been engaged in the production of light alloy shells for sea mines and large storage and other vessels in pure aluminium. We saw mines of various types in different stages of manufacture and were able to obtain a very full account of their production procedure. Schmiddings specialise in oxy-hydrogen welding since, they claim, this gives less porosity than other types of flame. Some arc welding, however, is carried out on unstressed parts.

The aluminium alloy used in the production of mine shells was one containing 2% Magnesium and 1½% Manganese, and is known as KSS. This alloy requires rather specialised technique and therefore receives separate treatment.

Mine Shells in KSS

The alloy is used in the soft condition, in which state it has a tensile strength of about 15 tons/sq.in. The shells varied in thickness from 7-10 mms. and it appeared that the most popular size was a roughly spherical mine about 4 ft. diam. with 8 mm. wall thickness. The sphere is built up of about 4 parts comprising 2 segments, a cap and a central belt. The segments are pressed cold in a number of operations on a friction press. The edges which are to be welded are then chipped or machined to the required chamfer, the type of chamfer depending on whether the welding is to be done on both sides or on one side only. Wherever possible runs are put on both sides in which case the edge preparation is double-v, but for the final weld, welding from one side can be employed.

Prior to welding all the parts are treated in a bath of caustic soda solution followed by dilute nitric acid. Welding is carried out using an oxy-hydrogen flame and filler rod of similar composition to the parent metal. We noted that many welders made a practice of finishing the weld by tailing off the run into the parent metal at an angle to the deposit, and we were informed that this reduced the possibility of shrinkage holes and cracks. From later conversations and our own experience, however, we doubt whether this is necessary.

The fluxes used were Firinit and Autogal A and N, the last mentioned for positions where residues could not be completely removed.

Generally speaking the welding was good, although where it was carried out from one side only the penetration was very erratic. There was evidence, however, of slight blistering in the parent metal adjacent to some welds.

After welding and preliminary removal of the residues in a cold water swill, the parts were treated in a bath of hot caustic soda solution followed by a dip in cold dilute nitric acid and a wash in cold water. The caustic soda and nitric acid were contained in brick-lined, steam-heated, tanks.

We noted that arc welding had been carried out on some internal stiffeners. The deposit was very porous indeed but this was not important as this weld carried no load. Electrodes used in this welding were made by Hermann Fliess.

As a rule no mechanical work was carried out on the finished welds although in certain cases the whole job was machined down to conform to some particular gauge.

Tests applied to the finished structure comprised X-ray examination of a proportion (usually about 10%) of all welds, and external pressure tests on all mines.

Pure aluminium vessels

The pure aluminium vessels which Schmidding produced were

mainly for the storage and transport of milk, petrol, and nitric acid. At this factory they produced no containers for hydrogen peroxide.

There were no special points of technique except, perhaps, their insistence that oxy-hydrogen gave the best weld. All welds in pure aluminium vessels were hammered and where special corrosion resistance was required, subsequently annealed.

This factory had carried out some tests on the welding of the high magnesium alloy Hy 75, but we were informed that the bulk production in this alloy was confined to their Bodenbach Works.

Persons interrogated

Dr. Ing. Bossert.	Works Manager.
Dr. Ing. Kleinert.	Director.

1.2. Wilhelm Schmidding, Hanover-Linden

This factory was engaged on similar work to the Schmidding factory at Köln-Niel. In addition to the mine-shells in KSS alloy and storage and transporttanks for milk and nitric acid, however, they had produced vessels in 99.5% aluminium for handling hydrogen peroxide and in KSS for liquid oxygen.

All the aluminium including the KSS alloy was produced by the Vereinigte Metallwerke in the next building. These latter people appeared to have facilities for producing very big sheets since we noted plates in pure aluminium and KSS about 22' x 9' x $\frac{1}{4}$" and 30' x 6' x $\frac{1}{4}$" in Schmidding's main shop.

As at Köln, oxy-hydrogen was considered to be the best welding flame for aluminium alloys. It was said that in addition to giving a better weld as indicated by X-ray examination, this flame, since it was cooler than oxyacetylene, was handled more easily by trainee welders.

Their lay-out for producing mine-shells was very similar to

that at Köln. They had, however, a rather wider range of types at this factory and appeared to tackle the more difficult and heavier sections. For instance we saw rings up to $1\frac{1}{2}$ in. thick which had been welded up from strip. These heavy sections, particularly when in cast alloy, required very thorough preheating before welding if cracks were to be avoided. Welding was usually carried out in a number of runs from one side whilst a preheating flame was held underneath.

Again metallic-arc welding was employed in certain parts of the internal structure of the mines but not in parts carrying stress. Full preheating was always employed before welding with the arc.

Certain other vessels for handling liquid oxygen had been made in $\frac{1}{4}$ in. thick KSS. These vessels were of simple cylindrical shape of about 1500 gall. capacity with domed ends. The welds were made by putting a run on each side as in the case of the mines. Internal baffles were fillet welded directly on to the shell.

Although as noted above most welding was carried out using oxy-hydrogen, atomic hydrogen was often used in repair. It was stated that since the atomic-hydrogen flame was hotter it gave less distortion than oxy-hydrogen and obviated the need for general preheating, where a small local weld was required.

Mines (and certain other vessels) were examined radiographically. A proportion (about 10%) were so examined and the quality of the welding revealed in this way was used as a basis for establishing wage rates. A good welder would be paid a substantially higher rate than a poor one. In addition of course, all mines were subject to a test under external pressure.

As regards vessels in pure aluminium, again there were no special features in construction and fabrication. For pure aluminium vessels which were to be used for storing and transporting hydrogen peroxide, a rather elaborate cleaning and waxing treatment had been specified but experience had shown that most of this was unnecessary, with the peroxide which was being produced towards the end of the war. They had found that provided the material

surface was thoroughly cleaned with strong nitric acid followed by washing in clean water, waxing was quite unnecessary. (See visit to Walterwerke, Kiel).

It is of interest to note that these people were making welded buckets and other small containers out of clad Duralumin. This, however, appeared to be an emergency measure and they were fully aware of the dangers from corrosion and weak welds.

Person interrogated

Herr Kraus - Works Manager.

1.3. Carl Canzler, Düren

Canzlers were no longer producing on this site. The equipment was compulsorily evacuated in September 1944, the various sections going to the following places:-

Chemical Plant	Otto Schickert, Rhumspringe and Bad Lauterberg.
Aluminium Transport Tanks	Rudisleben, near Arnstadt.
Clad Steel	I.G. Farben Leverküsen, Near Düsseldorf.
Welding of Plastics	I.G. Farben Wolfen, near Bitterfeld.

In the past, Canzlers have fabricated plant in all the usual non-ferrous alloys. More recently they have embraced the welding of clad steels and plastics. During the war, however, they have confined themselves to various grades of pure aluminium, KSS, and clad steels.

Generally speaking they do not appear to have produced large numbers of any one component (c.f.Schmidding) but have continued as jobbing copper-smiths. Most of their work has been in pure aluminium in which they have produced tanks for hydrogen peroxide, nitric acid, petrol and milk.

The information supplied by Heinrich Canzler is summarised under various headings below.

Welding Methods

The normal method of welding aluminium and its alloys was by oxy-hydrogen or oxy-acetylene flame. Canzler had no particular preference for any one of these two flames. (c.f. Schmidding) A certain amount of arc welding was employed, for instance in attaching brackets etc., or where freedom from distortion was important. In arc-welding it was essential to use a backing bar to control the penetration but preheating, Canzler said, was only necessary before laying down the first electrode. Canzler had produced one or two small vessels which were completely arc welded but it was too early to say how they had performed in service. Trials had been made, but without success, with automatic arc welding on aluminium using the Kjelberg head. They normally preferred electrodes made by Lauterwerke and Fliess.

Atomic hydrogen welding was not used since it offered no particular advantages over gas welding and the torch was more cumbersome.

The standard method of finishing welds was to hammer and, where corrosion resistance was important, subsequently anneal.

Welding of Pure Aluminium

There was no important developments in welding pure aluminium. As far as vessels for hydrogen peroxide were concerned Canzler stressed the apparent need for a very clean finish on the inside surfaces.

We were interested to hear that a code for pressure vessels was in operation. Canzler had a great deal to do in drafting this code. A copy was evacuated.

KSS and other magnesium-containing alloys

Canzler considered that the magnesium-containing alloys were

difficult to weld. The difficulty increased with increasing magnesium content, the worst trouble being cracking. They had produced some containers in 7% Mg. for T.V.A. Kiel before the war but since then they had standardised on KSS for most of their work requiring a strong alloy.

Duralumin

Dural-type alloys had been welded but the welds were weak and had poor corrosion resistance.

Testing

Canzlers carried out X-ray examination on all welds in pressure vessels. This was apart from the usual tensile and other tests required by the boiler code.

Person Interrogated

Heinrich Canzler - Managing Director.

1.4. Otto Schickert & Co., Bad Lauterberg and Rhumspringe

Canzler's equipment, which had been evacuated to these works from Düren, was examined. Most of this was for fabricating clad steels but some light gauge aluminium welding had also been carried out.

The Schickert Works at Lauterberg had been engaged in producing strong hydrogen peroxide. Those at Rhumspringe were intended for the same purpose but were never completed. Apart from Canzler's equipment they were of interest to us because of the large amount of aluminium equipment used in handling the hydrogen peroxide.

At Lauterberg the strong hydrogen peroxide was stored in welded aluminium tanks. Originally 32 vertical vessels 3 metres diam. x 5.5 metres high (35 cu. metres.) had been used. These had been built by Widmann of Mannheim and Schmidding of Hannover and their interior surfaces had been fully treated by cleaning and waxing.

Later they were supplemented by 12 large horizontal vessels 3 metres diam. x 13 metres long x 10 mm. thick (80 cu. metres). These latter vessels were not waxed on the inside. The metal used in all this work was of 99.5% minimum purity.

There were no special features about these vessels except, of course, their size.

In addition to its use in these vessels, aluminium had also been widely used in the pipe-work carrying the hydrogen peroxide, and in condensers. Again there were no special features about the construction.

At Rhumspringe too, aluminium was being used in very much the same applications as at Lauterberg.

Person interrogated

Dr. Schubert.

1.5. Ziemann A.G., Ludwigsburg near Stuttgart

It was known that this firm manufactured storage and rail transport containers, especially for concentrated hydrogen peroxide and liquid oxygen.

Materials Used

The bulk of the containers were fabricated in pure aluminium (99.5%), but Hydronalium 3 and 5 were also used frequently. Hydronalium 7 was used originally, but had been abandoned due to welding difficulties. KSS was extensively used for mine shells and small containers for torpedoes.

Shop Lay-out and Welding Procedure

Contrary to what was found at the various other places visited, this firm was laid out to produce containers on a large scale and, as far as possible, on the conveyor system. The shop lay-out allowed

for a proper flow of the components. One production line prepared the material for the outer hulls, whilst on another line the ends were cut, dished and shaped ready to be fitted into the outer hulls. Dishing of the ends was done by a series of electrically driven hammers which could handle discs up to 7 m. diameter. A great number of manipulators, jigs and welding fixtures were employed to facilitate the work. A special jig which was noticed was an anvil on a column with rollers on the end which could be placed inside the cylindrical part of containers and was intended to support the shell during the hammering of the weld seam; the distance between the rollers and the length of the column could be adjusted, thus permitting the use of this tool on containers of various diameters.

The firm has specialised in metallic arc welding and, wherever possible, all welding was done with this process. Where flame welding was still used oxy-acetylene was employed instead of oxy-hydrogen because of the cost. The latest type of container was, however, completely arc welded in the main seams of the shell. The thickness of the material varied between 8 and 40 mm., and it was pointed out that thicknesses up to 25 mm. were welded without copper backing plates in one run. The electrodes used were "Veral" rods manufactured by Wiener Vereinigte Metallwerke. This information seems to indicate that the manufacture of these rods had been transferred from Bitterfeld to Vienna and, as this rod is apparently the best one produced in Germany, it might be of interest to visit the Vienna factory.

Oxy-acetylene welding was still used on thicknesses from 3 to 5 mm. and the fluxes preferred were Autogal A and M, manufactured by I. G. Farben.

Atomic hydrogen welding had not been used because the firm mainly specialised in thicker material, but Mr. Wirth thought that this process was advantageous on thin sheets.

From the interrogation, it appeared that the firm had tried to weld aluminium automatically by means of the Kjelberg Head, but the results were not entirely satisfactory.

After welding, the reinforcement was chiselled off and the

seams were hammered cold to increase the strength and corrosion resistance. Several large containers which had been brought back for repairs after bomb damage were inspected, and it was found that all the welding seams had stood up perfectly although the deformation of the tank was very severe. It appears that no special surface treatment was carried out on the hydrogen peroxide containers, apart from anodising.

Testing

All containers and sea-mine shells were pressure tested and occasionally X-ray inspected. The X-ray tests were also applied for the grading of welders. No metallographic testing equipment was available and this type of investigation, especially for new developments, was carried out for the firm by the University of Stuttgart.

Persons interrogated

Dr. Schlaich - Technical Director and Works Manager.

Dip.Ing. Wirth - Welding Engineer.

1.6. C.A. Haraeus. G.m.b.H. Hanau.

Up to 1943 the main products manufactured by this firm were tanks and containers in steel and vessels for benzene and similar substances in aluminium, after this the firm changed to more general engineering. The interrogation of Haraeus and Ackermann concerned their knowledge of light alloy welding.

Pure aluminium (99.5%) was used for the large containers while some of the smaller ones, together with valves, were fabricated by welding from Silumin, Pantal and, at the beginning of the war K.S. Seewasser. No al.-Mg. alloys nor Alclad were used; neither metallic arc nor spot welding were employed the majority of the work being carried out with the oxy-acetylene process because of its cheapness and the high temperatures obtainable. Atomic hydrogen welding had been used for some small aluminium containers made for I. G. Farbenindustrie and Lwigi at Frankfurt.

Filler rods of the same composition as the parent metal were deposited and the latter was specified by the customers. Sheets up to 8 mms. in thickness were prepared for welding with a single Vee, above this a double Vee was used. Preheating was carried out where possible from the reverse side of the work by the application of a flame ahead of the welder. For double Vee preparations two operators would be employed on each side simultaneously. Containers with a wall thickness of 0.75 to 1.5 mms. were atomic hydrogen welded, above this gas was used.

Flux was originally obtained from Griesheim Autogen (I.G. Farbenindustrie), later from Guilini at Ludwigshafen and finally was manufactured by themselves to the following composition:-

Calcium chloride	($CaCl_2$)	68.7%
Lithium "	(LiCl)	18.7
Sodium fluoride	(NaF)	8.8
Sodium sulphate	(Na_2So_4)	3.8

Lap welds were at one time made by hand or pneumatic hammering. The sheets to be so joined were wire brushed before being preheated to 450°C; no flux was applied and the temperature was raised to about 650° and hammering was continued until it fell to 450°C. 100 mms. overlap was usual practice.

Pressure testing only was applied to the finished vessels of all kinds and cracks or flaws found were cut out and rewelded. No X-Ray examination was undertaken. The welders were tested periodically as laid down in the regulations for pressure vessels.

It was considered that this firm offered nothing new or outstanding so far as the welding of light alloys is concerned.

Persons Interrogated

Dr. R. Haraeus - Director
Herr Ackermann - Engineer

1.7. F. Widmann u. Sohn, Neckarau, near Mannheim

This firm is old established and was the first German concern to weld aluminium in 1907. Storage vessels of all kinds have been made chiefly from pure Aluminium (99.5%) and Silumin for light alloy work although, in certain instances, where specified by the customer, Hydronalium (3% mg) and K.S.Seewasser have been used. Latterly composite tanks had been a speciality; these consisted of an internal container made of pure aluminium 2 mms. in thickness, welded by the oxy-hydrogen process and an external steel container also welded. The aluminium tank was made with a lip welded on mid-way along its length; this was dished inwards before the end of the tank was welded in place to enable it to be inserted in the steel protector tank. When in place the lip was hammered out through the corresponding hole in the steel shell, and the end of the aluminium tank welded on followed by the welding of the end of the steel tank. This enabled the lip to be of aluminium so that liquid being poured in or out would not come in contact with the steel and also the two tanks to be a good fit, one within the other.

The largest aluminium tanks that the firm had made were of 300,000 litres capacity for I.G. Farbenindustrie, Bokum.

Flame welding was used exclusively for the fabrication of the aluminium containers themselves and the oxy-hydrogen process was preferred. It was stated that the oxy-acetylene flame was more difficult to use, being too hot for the thinner aluminium sheet and the gas, as obtained from generators, was not pure enough.

Impurities in the gas tended to produce harder weld metal and cause the entrapment of inclusions in the deposit. Filler rods were of similar material to the parent metal and "Autogal" fluxes only were used. The thickest material welded normally was 40 mms. but occasionally material up to 65 mms. in thickness for vacuum vessels had been fabricated. The sheet was prepared for welding with a double Vee if it were over 8 mms. in thickness and two operators welded vertically upwards from both sides simultaneously. Preheating with a flame was always carried out on material of over 20 mms. thickness and the weld was hammered subsequently until cold. Tanks which were to contain nitric acid were annealed after welding

to increase the corrosion resistance of the weld, the critical position for such corrosive attack being the junction of the weld and parent metals.

Metallic arc welding was only used for fillets, brackets, lifting eyes, and for fixing cooling coils but not for the main runs of the tanks. The largest rod employed was 6 mms. diameter for material 12 mms. in thickness. Atomic hydrogen welding was not used.

Hammer or pressure welding had only been tried experimentally on aluminium but other materials such as nickel were joined in this way.

Any testing of the welds or metallurgical examination was carried out for the firm by I. G. Farbenindustrie at Ludwigshafen. This section of the I. G. concern produced a substance called "Nickal" which was specially suited for the detection of small leaks in vessels. This is in powder form to be mixed into a paste with water and painted over the area (such as a welded seam) to be tested. With an air pressure of $\frac{1}{2}$ atmosphere cracks or porosity could be located by the formation of bubbles by the paste. It was stated to be much more effective than a soap film, a small sample of the powder was procured.

During the course of the interrogation, mention was made of a method the firm used for protecting the threads of aluminium spouts, bosses and taps on the tanks which were made. The process was known as "Granitierung" was carried out by the firm of Leuchner of Berlin and now located in the works of Otto Schickert at Bad Lauterberg. The process was not anodising but imparted a relatively hard and wear resistant surface to any articles so treated. Anodising was, however, placed out to contract for small parts where necessary.

Person interrogated

Herr Franz Heller.

2. REPORTS ON VISITS TO ELECTRODE AND FLUX MANUFACTURERS

2.1. Hermann Fliess and Co., Duisberg

This firm's production of aluminium electrodes ceased in 1944 due to bombing. Before this time they had made a range of dipped electrodes for welding pure aluminium, Silumin, Hydronalium and K.S.Seewasser. In all cases the core wire was of the same composition as the material to be welded and the coating was stated to be always of the following composition:-

Lithium Fluoride	10	parts	by	weight
Potassium chloride	45	"	"	"
Sodium chloride	30	"	"	"
Cryolite	20	"	"	"
Sodium sulphate	0.2	"	"	"

Electrodes were tested by making a butt weld and carrying out tensile and bend tests on specimens cut from it. Any metallographic examination required was carried out at the Schweiss Technische Lehr und Versuchsanstalt in Duisberg.

This establishment was located and Direktor Hase was interrogated; no information on aluminium welding was obtained but the system used for training welders evoked some interest and favourable comment.

2.2. Citogen A.G. Metallbearbeitung, Düsseldorf

After visiting several places to which the firm had successively evacuated the firm, now known as Cetto & Co., was finally contacted by telephone. It was stated that they now manufactured only a very limited quantity of dipped mild steel electrodes. Aluminium electrodes were no longer made and further investigation was therefore considered unnecessary.

2.3. I. G. Farbenindustrie - Griesheim Autogen, Griesheim, near Frankfurt.

This firm was one of the leading manufacturers of gas welding equipment, including fluxes. Samples of four types of flux for welding and soldering aluminium were obtained and are being analysed.

In reply to questions concerning aluminium alloy welding practice it was stated that oxy-acetylene welding was usually favoured for thick material and oxy-hydrogen for thin material. Pure aluminium and K.S.Seewasser were the most readily weldable materials and of the higher strength alloys Hydronalium with up to 5% magnesium could also be welded. With 7% magnesium, however, cracking troubles were encountered and the 9% magnesium alloy had been found definitely unsuitable for welding. The "Mausi" effect was discussed and it was considered to be entirely due to the presence of hydrogen in the parent material; the addition of small quantities of zinc and manganese to the aluminium-magnesium alloys in which this trouble arises was understood to reduce the severity of the effect.

Metallic arc welding was considered to be advantageous for fillet welds and seam welds in pressure vessels and because of this work had been carried out to produce an improved arc welding electrode. Satisfactory progress had been made in developing a non-hygroscopic coating which was to be applied by extrusion, but its composition had not been finally settled.

Persons interrogated

Dip. Ing. Bike.
Dr. Dümpelmann.
Dr. Ing. Holler.
Dr. Maier.

3. REPORTS ON MISCELLANEOUS TARGETS

3.1. Walterwerke, Kiel

Propulsive devices developed by this firm employed highly concentrated hydrogen peroxide (Ingolin) as the oxygen carrier. The purpose of our visit was to find out what welding methods were used in fabricating containers for Ingolin, what materials were used and how they were protected.

Storage Tanks

Ingolin storage tanks and containers for various purposes were obtained from several outside manufacturers, in particular Schmiding, Bodenbach.

10 storage tanks were installed, each of 1,500-2,000 gallon capacity (approximately 7 ft. diameter x 10 ft. high). A number of smaller transport tanks holding approximately 200 gallons were also examined together with several small containers similar in general design to milk churns and holding about 10 gallons.

The material employed was 99.5% purity aluminium approximately $\frac{1}{4}$" thick. They had been welded by the oxy-acetylene or oxy-hydrogen process and the welds hammered, the inside seams being also ground and polished. There was no special feature about these storage tanks apart from the necessity to have a good surface finish wherever Ingolin was in contact with the material. The tanks were not welded at these works and, in fact, the only light alloy welding carried out here was in the fabrication of pipe work used in the storage section. Oxy-acetylene welding was used for this and the material appeared to be 99.5% aluminium.

Details of the methods of treating the surface were obtained. The process laid down consisted of thorough cleaning of the inside surface with a 8-10% solution of caustic soda or a proprietary alkaline degreaser known as "P.3". Following this, the inside surfaces were treated with 1:1 nitric acid for one hour, followed by thorough washing using water with an iron content not greater than 0.1 milli-

gramme per litre. Originally, the surfaces were then waxed, but it was stated that waxing was no longer required due to the increased purity of the hydrogen peroxide.

Fittings

Valves and other fittings in contact with Ingolin were of "Hydronalium 5" or "Hydronalium 7" either machined from wrought stock or forged. Other fittings were of a 3% Mg. alloy in the cast condition or were of "Silumin beta" or "Silumin gamma". Fittings were generally anodised by the oxalic acid process.

Pumps

Pumps for handling Ingolin consisted of a 3% Mg. alloy cast casing or a "Silumin gamma" casing, with 13% chrome steel impeller and shaft.

Pipe Work

Pipe work was of 99.5% aluminium, oxy-acetylene welded, or of welded polyvinol-chloride which was said to incorporate a special plasticiser developed by I. G. Farbenindustrie. The hot air torch used for welding p.v.c. fittings was demonstrated.

Containers

For the containers employed in various projectiles, aluminium-magnesium alloys were used and again the welding was carried out mainly by Schmidding. The alloy was stated to be a special development of "Hydronalium 7" in order to avoid the so-called "Mausi" effect in the vicinity of the weld. ("Mausi" was a code word for a particular projectile).

Von Tinti explained that the Mausi effect was the blistering and porosity which developed alongside the weld in aluminium-magnesium alloys and that a new alloy had been developed by Dr. H. Röder of the Leipziger-Leichtmetall-Werke in collaboration with von Tinti and Messrs. Schmidding. This alloy is variously known as Hy 75 or Hy 7S (S standing for "special") made by the Leipziger-Leichtmetall-Werke or Hy 7K by I. G. Farbenindustrie.

Development of Special Weldable Alloy

Steps in the development of this alloy were outlined as follows:-

The mechanical properties required were those of the 7% Mg. alloy, but the magnesium content was reduced to 5.6% in order to increase weldability.

In order to restore the strength so reduced, the manganese content was increased to 0.8%.

The blistering was stated to be due to the gas content of the parent metal and special measures were stated to have been devised for obtaining gas-free ingots; in particular the silicon content was increased from 0.2 - 0.3% up to 0.4 - 0.5%.

Zinc was introduced to the extent of 0.4 - 0.5% and was intended primarily to (i) prevent precipitation of the beta constituent at grain boundaries, (ii) reduce tendency to grain growth in the over-heated zone; these two alterations improving the resistance to corrosion. The desired composition was stated to be as follows:-

Magnesium	5.6%
Manganese	0.8%
Silicon	0.5%
Zinc	0.5%

The minimum ultimate tensile strength specified for this material was 31 kg/mm^2 with an elongation of 10-16%, it being stated that actual material gave tensile strength figures of 38 kg/mm^2. The preferred composition for the corresponding 3% Mg. alloy (known as Hy 35) was as follows:-

Magnesium	3.2-3.8% (3.5)
Silicon	0.5-0.8% (0.6)
Manganese	0.3-0.6% (0.45)
Iron	0.5% max.
Copper	0.1% max.
Zinc	0.2% max.

In addition to the attention paid to composition, it was stated that each batch of material produced was submitted to a series of tests, by arrangement with Schmidding, in order to determine the suitability of the particular batch for fabrication by welding. These tests include X-ray examination, full mechanical tests (including impact tests) and examination of sections of welded material. The material was not only required to pass these weldability tests, but the tests also enabled the suppliers to specify the method to be employed for welding a particular batch of material, i.e., oxy-acetylene, oxy-hydrogen, or atomic hydrogen. Von Tinti stated that the majority of welding was in fact carried out by the oxy-acetylene process.

Protection of aluminium-magnesium alloys for Ingolin containers

It appeared that the aluminium-magnesium alloys were less resistant to corrosion by Ingolin than pure aluminium and they were therefore anodised. The W.X. procedure was used for welded parts and the G.S. process for turned parts.

(NOTE: The W.X. Process is - alternating current, oxalic acid.
The G.S. Process is - direct current, sulphuric acid.)

After anodising, the parts are then dipped in molten wax of the paraffin-type and are placed in an oven at the required temperature, the surplus wax being removed by wiping with chamois leather. Attempts to coat the containers with wax by means of a solution of wax in white spirit were unsuccessful as the evaporation of the spirit resulted in cracks or pores in the coating.

It is considered important to follow up with the Leipziger Leichtmetall Werke and I. G. Farben Industrie the further development of the high strength weldable aluminium-magnesium alloy referred to above.

Persons interrogated

Baron von Tinti	Metallurgist
Herr Kruske	Purchasing
Dr. Dietrichsen	Assistant metallurgist
Dr. Hausberg	Engineer

3.2. T.V.A. (Nord) - Torpedo Works - Eckernforde

Torpedo components were obtained from various factories elsewhere and only assembly operations were carried out here. A small amount of work had been welded, principally by the oxy-acetylene process for which standard Griesheim and Messer equipment, including small low pressure generators, was available. Standard "Autogal" and "Hydrogal" fluxes were used, but there appeared to be no specific information available on the welding of the aluminium-magnesium alloys.

Metallic arc welding of aluminium alloys had not been carried out but a new Kjelberg automatic welding plant with twin electrodes was examined. It had been used only as a normal motor-generator welding set for steel and should prove of interest in this country. It was recommended that it should be evacuated, particularly as the Kjelberg works are now in the Russian zone.

Two un-used Ingolin containers approximately 7ft. diameter x 8 ft. deep were examined. They had been fabricated from $\frac{1}{4}$" thick, pure aluminium by Messrs. Gebr. Böhling (Hamburg) and appeared to be normal in so far as welding was concerned. A single horizontal cylindrical storage tank was also examined. It was of about 500 gallons capacity and appeared to contain hydrogen peroxide. There were no special features apparent.

Person interrogated

Herr Kämpe - Welding engineer

3.3. Prof. A. Matting, Technische-Hochschule, Hannover

Dr. Matting is Professor of Materials, and was, of course, very well-known before the war in connection with welding. He stated that he was still particularly interested in welding and discussed latest developments in gas, arc and resistance welding processes. He had been evacuated to Lüneburg and was only just returning his documents, etc., to the habitable portions of the Technische-Hochschule.

In connection with aluminium alloy welding, he confirmed that considerable difficulty had been experienced with aluminium-magnesium alloys in particular the "Mausi" effect, and he considered that this was related to the gas content, in particular, hydrogen. He was aware of the work of Dr. Mäder and had correspondence in his files with Mäder. He stated that, recently, manufacturers of aluminium-magnesium alloy sheet had successfully reduced the gas content of their materials, but he appeared to have no details of the methods employed. Some reference was made to this problem in the new edition of his (Matting's) textbook on light alloy welding ("Anleitungsblätter für das Schweissen und Löten von Leichtmetallen", published 1940 in Berlin by V.D.I. Verlag. G.m.b.H.) Typescript copies of a number of revised chapters of this book were evacuated.

A copy of a report by Herrmann of Junkers on pressure welding of light alloys was also removed from his files, although he was unable to give any details about this work. An abstract of this important paper is given in the Appendix.

Matting had also edited the journal "Elektroschweissung" which ceased publication with issue No. 10, Vol. 15, 1944. Copies of Vols. 14 and 15 were evacuated. Copies of the following three papers were also evacuated:-

1. (Copy of printed paper) "Uber mechanische und physikalische Eigenschaften von Rein-Al und einiger Al-Legierungen bei der Temperatur des Flüssigen Sauerstoffs". H. Mäder (Rackwitz).

2. (Typescript) "Die Kerbschlag-zähigkeit von Leichtmetall-Schweissverbindungen". P. Brenner und A. Matting.

3. (Photostat) "Entstehung, Entwicklung und Eigenschaften der Legierung Hy 35". (I.G. Farbenindustrie).

There is no doubt that Professor Matting had a very considerable amount of information on welding not only of light alloys, but of other non-ferrous metals and steels.

3.4. Verinigte Leichtmetall-Werke, Hannover

Dr. Schulze and Dr. Brenner were questioned concerning aluminium alloys suitable for welding. They stated that during the war the firm had supplied W. Schmidding and other fabricators with pure aluminium and K.S.Seewasser for work on which welding was required. The alloy K.S.Seewasser was stated to have better resistance to corrosion by seawater than commercially pure aluminiun, its strength in the annealed condition is about 15 tons/sq.in. and its weldability is good. It was manufactured to the following composition:-

Copper	< 0.05
Manganese	0.6 - 1.0
Magnesium	2.3 - 2.8
Silicon	< 0.8
Iron	< 0.5
Zinc	< 0.1

Also during the war the firm had been required to make the alloy Hy35 (3-3.5% Magnesium, about 1% Manganese 0.7 - 1.0% Silicon) which had been developed by another firm. It was stated that thick welded vessels had been made from K.S.Seewasser and thinner vessels from Hy35. Discussing this latter alloy Dr. Brenner said that with magnesium contents greater than 3.5% the weldability of the Al-Mg alloys decreases because of blistering at the weld. The firm had apparently carried out no work to develop a weldable alloy having a higher strength than the alloys mentioned.

This firm manufactured the well known series of "Veral" electrodes for arc welding aluminium and a wide range of its alloys. Nine types were produced as follows:-

Electrode	For Welding
Al-Cu-Mg.	Duralumin, Alclad.
Al-Cu-Ni.	Y Alloy.
Al-Cu.	Lautal.
Al-Mg-Si.	Anticorodal
Al-Mg-5.	Hydronalium, B.S.Seewasser.
Al-Mg-Mn.	K.S.Seewasser.
Al-Si-5% }	Silumin
Al-Si-12% }	
Al-Mn.	Aluman.

In all cases the electrodes gave deposits of approximately the same composition as the alloys on which they were intended to be used. However, it was evident that the aluminium-silicon electrodes had been used more extensively than the above list indicates. For instance, at the time of our visit the firm were themselves making some large window frames by arc welding extruded duralumin sections, using an aluminium-silicon electrode. The joint was preheated with a blowpipe flame before arc welding.

4. SUMMARY

It will be seen from the preceding reports on individual firms that the majority of the German thick section light alloy containers were made from pure aluminium or K.S.Seewasser. Pure aluminium vessels were used for the storage and transport of milk, petrol, nitric acid and concentrated hydrogen peroxide, that is aluminium was used, as in this country, in cases where strength was not very important or where maximum corrosion resistance was required. K.S.Seewasser was used where greater strength, coupled with a considerable degree of resistance to corrosion, was necessary, for instance large numbers of mine shells were made from this alloy. Its composition appeared to vary slightly from one manufacturer to another, typical compositions being 2% magnesium, 1.5% manganese and 2.5% magnesium, 0.8% manganese and usually the alloy contained up to 0.8% silicon. It is of course readily weldable and it has a strength in the annealed condition of about 15 tons/sq.in.

The difficulties encountered in trying to weld the stronger alloys containing high percentages of magnesium were widely known. Two of the six fabricating firms visited stated that they had made welded containers from the 3% magnesium alloy and one of these two firms had also welded the 5% magnesium alloy on a production scale. Attempts to use the 7% magnesium alloy for welded parts had been made by several of the firms but they had proved unsuccessful, mainly because of the so called "Mausi" effect. This was the name given to the blistering which occurs adjacent to the welds with consequent reduction in strength. It was realised that this blistering is associated with a high parent metal gas content and that the effect becomes more severe with increasing magnesium content. One light alloy manufacturer, the Leipziger Leichtmetall-Werke had co-operated with designers and fabricators in an attempt to improve the weldability of the aluminium-magnesium alloys. The alloy resulting from this work was known as Hy75 or Hy7S, or when made by I. G. Farben as Hy7K, it contained 5.6% magnesium and was stated to have a tensile strength of 20 to 24 tons/sq.inch. It appeared to have been used on a fairly large scale for making containers for projectiles.

Work in this country by the B.N.F.M.R.A. has shown that the presence of minor impurities such as lithium, barium, strontium and particularly calcium in the aluminium-magnesium alloys greatly increases their tendency to develop porosity on welding, also that the furnace atmosphere used during processing influences the amount of porosity developed. Exposure to wet atmospheres promotes porosity while the co-presence of hydrogen fluoride in the furnace atmosphere appears to inhibit this effect. It has also been proved that material containing up to 7% magnesium does not develop porosity provided care is taken to degas in melting and to exclude the impurities mentioned previously and also that during processing the material is heated in electric furnaces, i.e., in ordinary air. Material of higher magnesium content develops porosity even when these precautions are observed, but if small additions of hydrogen fluoride are made to the atmosphere of the furnace used for annealing, neither the high magnesium nor the calcium containing material develops porosity on welding.

No evidence was obtained on this trip to indicate that German manufactueres appreciated the importance of either the alkaline earth metal content of the alloy or the annealing furnace atmosphere in connection with the blistering of these alloys on welding. But in this connection it is probably important to mention that a German welded container examined in this country during the war made from an aluminium-magnesium alloy containing 5.8% magnesium and that this material was found to be particularly good for welding, the weld boundaries being free from porosity. On being examined spectrographically the alloy was found to contain a small quantity of calcium and a larger proportion of lithium, but the quantities were lower than would normally be expected. This then rather suggests that the importance of reducing the amount of these impurities normally picked up from melting fluxes had been recognised.

Gas welding was normally used for all the important joints in the different types of aluminium alloy containers mentioned above. Three of the six fabricating firms visited used oxy-hydrogen welding in preference to oxy-acetylene because it was considered that the former process gives sounder welds, that the flame is easier to manipulate and that it was therefore more suitable for inexperienced war time welders. Two firms preferred

oxy-acetylene welding to oxy-hydrogen, mainly because of its lower cost. Only one firm used arc welding extensively for main joints, other firms used it only for lightly stressed welds and those not required to be pressure tight. When arc welding was used preheating was almost invariably employed. Atomic hydrogen welding appeared to have been used to a very small extent, either for repair work or for welding very thin materials.

The range of commercial gas welding fluxes available included a non-corrosive (presumably fluoride) mixture for use in making joints which could not be thoroughly cleaned after welding. At least two firms had manufactured arc welding electrodes giving parent metal deposits on a wide range of aluminium alloys.

Welds in thick materials for food and other containers were usually finished by hammering, and when corrosion resistance was particularly important the welds were subsequently annealed.

Four of the fabricating firms visited inspected their aluminium alloy welds by means of X-rays and the welders wage rate was usually based on the quality of his welding as judged by this method of inspection.

5. CONCLUSIONS AND RECOMMENDATIONS

German practice in fabricating thick walled aluminium and aluminium alloy containers differed in several respects from our own. Two of the most striking of these were the wide use of the alloy known as K.S.Seewasser, which up to the present has not been much used here, and also the use by several firms of oxy-hydrogen rather than oxy-acetylene welding. German firms have welded aluminium alloys containing as much as 5.8% magnesium on a production scale in spite of the fact that their knowledge of the fundamental factors involved in welding aluminium-magnesium alloys appeared to be less than our own. In the sphere of press welding too the Germans had used a process on a large scale (for making aircraft heat exchangers) without investigating its fundamentals nearly as thoroughly as we have done.

It is considered particularly important to follow up with the Leipziger Leichtmetall Werke and I. G. Farben the further development of the high strength weldable aluminium-magnesium alloy referred to above. A recommendation that non ferrous welding specialists should interrogate Professor Matting has already been acted upon, also another team has obtained additional information concerning the Junkers press welding process. Separate reports will of course be issued by these teams.

APPENDIX

Abstract of article "Press welding of aluminium and aluminium alloys", by H. Hermann, Dessau. (Translation by H. O. Willrich).

This paper describes a press welding procedure developed and used by Junkers Flugzeug und Motorenwerke A.G. The welding is carried out in the plastic state under high pressure but no melting occurs. In order to obtain a good joint a certain amount of deformation is necessary. The process is carried out by placing two thin sheets one on top of the other between the heated welding dies (fig.1A), heat is conducted from the tools into the sheets and when the necessary preheating temperature is reached, they are squeezed together in the press. The procedure was developed for making an aluminium alloy radiator part from two sheets of Pantal 0.4 mm. thick which were simultaneously pressure welded and formed by compressed air.

Welding dies

The best material for the dies is steel of the composition, carbon 1.8-2%, silicon 0.5%, chromium 12.3%, vanadium 0.45%, manganese 0.5%. A pair of dies made from this steel will make up to 750,000 welds in annealed Pantal. The lower die rests on springs the purpose of which is to press the two halves lightly together during the early part of the stroke and so conduct heat to the workpieces. At the bottom of the stroke the springs are completely compressed.

The press welded radiator part required a welding pressure of 120-130 tons, the length of the welded seam being 13 metres; the width of the seam is indicated in fig. 4 which is a section through one element of the part. The heating of the dies was done by pyrometrically controlled gas burners.

Compressed air for forming the parts was fed in by means of a special ring fitting which was placed between the sheets at a place where the dies were suitably recessed. The thickness of the ring was such that a pressure tight joint was formed between

it and the sheets during the welding and forming operation. The inlet valve for the compressed air was actuated by the press itself.

Welding time, temperature and pressure, strength and extent of deformation

The time of 30 seconds for making the radiator part from two 0.40 mm. sheets was made up as follows:-

5 seconds preheating time - towards the end of that period compressed air was blown in.

1 second welding time.

5 seconds interval for exhausting compressed air.

19 seconds for reloading dies.

The temperature at which aluminium alloys can be press welded varies with the composition of the alloy and is between 420 and 480°C; the required pressure lies between 300 and 800 kilogrammes/sq.cm. It is relatively low for annealed pure aluminium and is approximately 500 kg/sq.cm. for Pantal.

The strength of press welded joints depends on the extent to which the thickness of the material is reduced at the weld, that is on the amount of deformation necessary to make the weld. For annealed Pantal a deformation of approximately 50% is necessary but it depends on the time interval which elapses between cleaning the surfaces and carrying out the welding. The maximum time for this interval is 1 to 1½ hours.

Hard rolled sheets can be more easily welded than annealed sheets. The weldability of aluminium alloys by this process is as follows, hard rolled aluminium-manganese sheets and hard rolled pure aluminium can be most readily welded, followed by Pantal and aluminium-zinc-magnesium alloys. Hydronalium is definitely inferior and no satisfactory results have so far been obtained with Dural and Duralplat. Magnesium alloys cannot be press welded.

Cleaning of the sheets

The cleaning of the sheets is of great importance. This can best be done by the following procedure:-

1. Dip for 30 secs. in 15-20% nitric acid at room temperature, rinse in water.

2. Dip for 30 secs. in 15-20% sodium hydroxide solution at 50-60°C, rinse in water.

3. Dip again in 15-20% nitric acid, rinse in water.

After drying the sheets are wire brushed on the inner side. Cleaning of the sheets by pickling or wire brushing alone is insufficient. The cheapest method successfully applied in mass production is to have the sheets pickled at the rolling mill prior to the last rolling process, then finished on polished rolls and to wire brush immediately before press welding.

Sticking of the sheets to the dies

In order to prevent the sticking of the sheets to the dies two things are necessary:-

(a) A certain roughness of the die-surface which cannot be properly achieved by machining. The method at present used is to grind the surface of the die and to sprinkle it with a mixture of talcum and potassium nitrate prior to the first heating, usually the first 6 to 10 welds stick to the dies and have to be torn away. After that the tool surface becomes roughened and finally the tendency to stick disappears completely.

(b) Every second part is brushed with a thin aqueous paste of talcum and potassium nitrate in the proportions 2:1, this has to be washed off immediately after welding to avoid corrosion.

Applications of the process

The process is most suitable for mass production, due to the necessity of using precision tools; built up dies have proved successful. The process lends itself especially to the production of hollow bodies or shells because the welding can always be accompanied by a forming process or some other means of avoiding distortion. Examples of the application of this process are heat exchangers, radiators and condensers, on which expensive tubes can be replaced by troughs formed in the sheet, toys, parts for motor bicycles and bicycles, for instance handlebars and headlamps. Steel parts can be pressed in or shrunk in. Other examples are double walls or partitions, doors for cupboards etc., which have to be produced in great quantities. Theoretically the thickness of the sheet is unlimited. So far sheets up to 5 mm. have been used; the heating times increase with the thickness.

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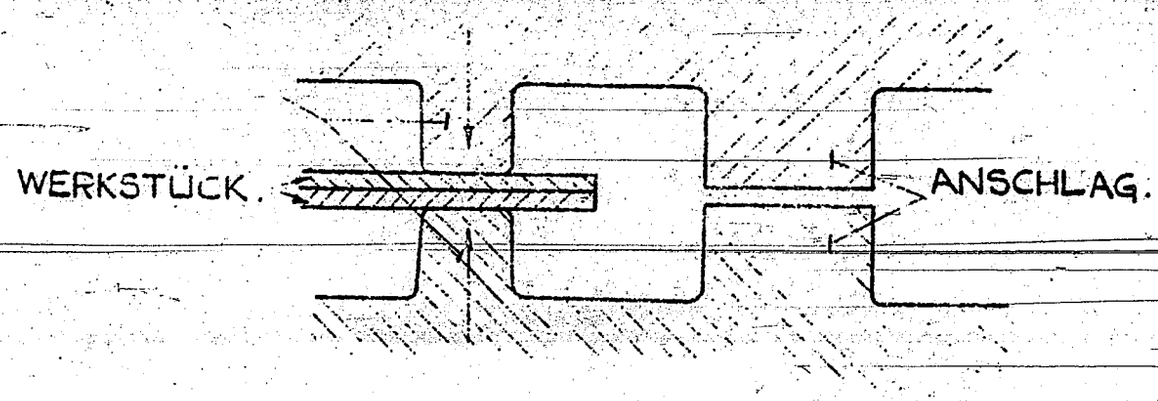

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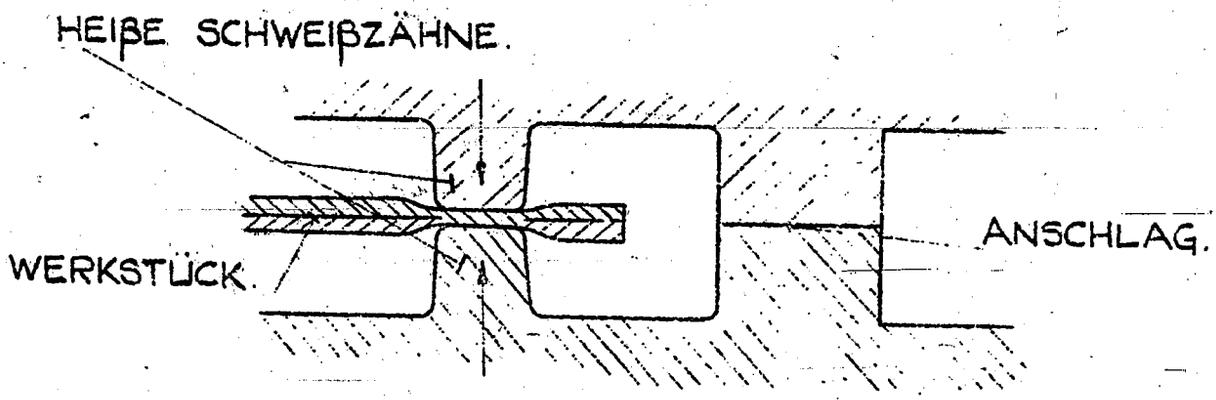

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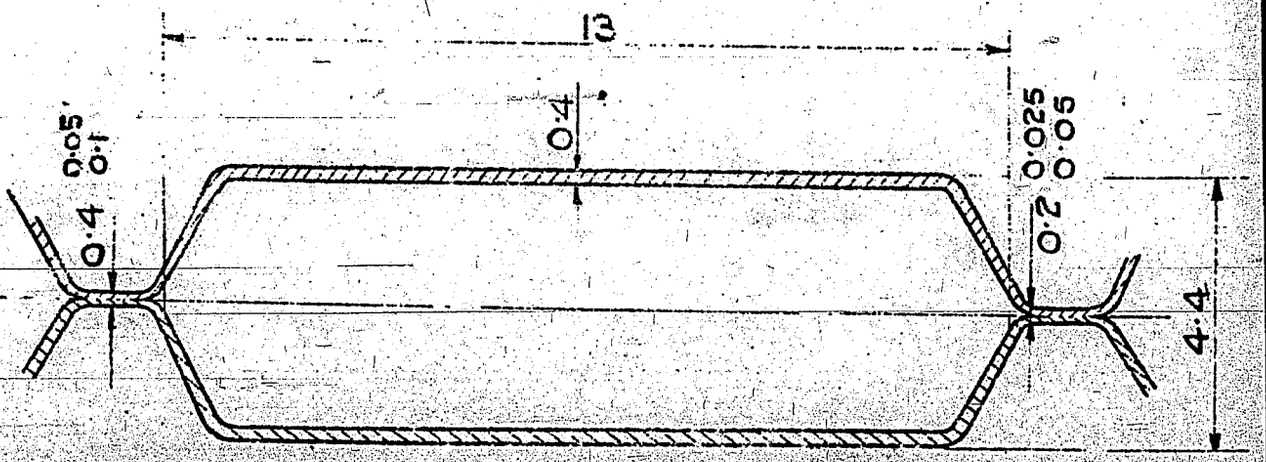

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FINAL REPORT No. 137 (Appendix)
ITEM No. 22

CYCLOPOLYOLEFINES (PAPER BY DR. J. W. REPPE, I. G. FARBEN RESEARCH CHEMIST)

APPENDIX

"This report is issued with the warning that, if the subject matter should be protected by British Patents or Patent applications, this publication cannot be held to give any protection against action for infringement."

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CYCLOPOLYOLEFINES

(Paper by Dr. J.W. Reppe, I.G. Farben Research Chemist)

Appendix – Cyclo Units

Translation issued by
B.I.O.S. Group 3

C.I.O.S. Item 22
(Miscellaneous Chemicals)

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20 p. diagrs.

APPENDIX TO REPORT BY W.J. REPPE ON CYCLOPOLYOLEFINES

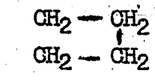
This Appendix giving a list of compounds mentioned in the text of B.I.O.S. Final Report No. 137 should be attached to the latter report.

A. E. CHILDS

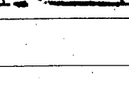
Chairman, Group III, B.I.O.S.

Appendix to Report by W. Reppe on Cyclopolyolefines

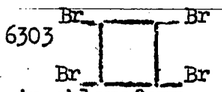
C_4H_8 — Cyclobutane



$C_4H_4Cl_2$ — 1.2 Dichlorocyclobutene 3.
Solid form. White plates from ligroin mp. 44°C: bp. 130°/760 mms.
Liquid form $n_D/20$: 1.5121 $D\ ^{20}/_4$: 1.2692
By thermal decomposition of the adduct of the dichloride $C_8H_8Cl_2$ with naphthoquinone or ester of acetylene dicarboxylic acid.

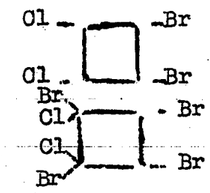


$C_4H_4Br_2$ — 1.2 Dibromocyclobutene 3.
Colourless oil. bp. 174-175°C./760 mms. By thermal decomposition of adduct of dibromide $C_8H_8Br_2$ with ester of acetylene dicarboxylic acid.



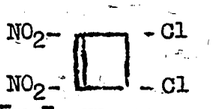
$C_4H_4Br_4$ — 1.2.3.4. Tetrabromocyclobutane.
Colourless oil. bp. 110°C/1.5 mms. n_{20}^{D} 1.6303 D_4^{20} 2.3675
Bromination of $C_4H_4Br_2$ with 1 mole. Br_2 in chloroform.

$C_4H_4Cl_6$ — 1.1.2.3.4.4. hexachlorobutane. $Cl_2CH\text{-}CH\text{-}CH\text{-}CHCl_2$ (Cl Cl)
Colourless prisms from ether, acetic acid or benzene. mp. 109-110°C.
By bubbling chlorine through a chloroform solution of $C_4H_4Cl_2$ at 25-30°C.



$C_4H_4Cl_2Br_2$ — 1.2 dichloro 3.4 dibromobutane.
Colourless liquid bp. 69°C./0.6 mms. n_{20}^{D} 1.5757. D 20/4 2.0784
By addition of 1 mole. Br_2 in chloroform

$C_4H_2Cl_2Br_4$ — Probably 1.2 dichloro 1.2.3.4. tetrabromocyclobutane.
Colourless prisms from acetic acid mp. 128-129°C.
By addition of 2 moles. Br_2 in CCl_4 to $C_4H_4Cl_2$



$C_4H_2O_4N_2Cl_2$ — probably. 1.2 dichloro. 3.4 dinitrocyclobutene 3.
Yellow prisms from CH_3OH mp. 127°C.

$C_7H_{12}O_4$ — Pimelic acid. $HOOC.CH_2.(CH_2)_3.CH_2.COOH$
White crystals from benzene mp. 104-105°C.
By oxidation of the fully hydrogenated product of the reaction between NaOMe and $C_8H_8Cl_2$

C_8H_{10} Cyclooctatriene (1.3.6)
Colourless liquid bp. 147-148°C./760 mms.
By treatment of the lithium derivative of cyclooctatetraene with alcohol.

C_8H_{14} Cyclooctene
Colourless liquid bp. 140°C/760 mms.
64°C/50 mms.
By hydrogenation of cyclooctatetraene in MeOH, dioxan, tetrahydrofurane in presence of $Pd/CaCO_3$.

Bicyclo (0.2.4) octane.
Colourless liquid bp. 138°C/760 n D/20 1.4613
D 20/4 0.8573
~~From $C_8H_{12}Cl_2$ by hydrogenation under pressure in~~ alkaline solution with nickel catalyst.

C_8H_8O Phenylacetaldehyde.
b.p.t. 184°C/760 mms.
1) From cyclooctatetraene with mercury salts.
2) From $C_8H_8Cl_2$ with water together with polymeric products.
3) From the oxide of cyclooctatetraene with dilute sulphuric acid.
Oxime. mp. 99-100°C, semicarbazone mp. 154°C.
phenylhydrazone mp. 57°C.

7.8 Epoxycyclooctatriene (1.3.5)
Colourless liquid bp. 73°C/12 mms. n D/20
1.539. D 20/4 1.063.
By action of perbenzoic acid on cyclooctatetraene in chloroform.

$C_8H_8Cl_2$ 7.8 dichlorobicyclo (0.2.4) octadiene (2.4)
Colourless liquid bp. 62°C/0.5 mms. n D/20
1.5417. D 20/4 1.2468.
1) By action of 1 mole SO_2Cl_2 on cyclooctatetraene in $CHCl_3$ or CH_2Cl_2
2) By action of calculated quantity of Cl_2 on cyclooctatetrene in $CHCl_3$ or CH_2Cl_2 at 0-30°C.

$C_8H_8Cl_4$ 2.5.7.8 Tetrachlorobicyclo (0.2.4) octene 3.
Solid form. Plates mp. 111-112°C.
Liquid form. Colourless oil bp. 126-128°C/1 mm.

$C_8H_8Cl_6$ 2.3.4.5.7.8. Hexachlorobicyclo (0.2.4) octane
Colourless plates from isobutyl alcohol mp. 126-127°C.
There is a liquid isomer. bp. 153-160°C/1 mm.

$C_8H_8Br_2$ 7.8 Dibromobicyclo (0.2.4) octadiene 2.4
Colourless liquid bp. 90°C/1 mm. n D/20 1.5951. D 20/4 1.7755.
By action of 1 mole. Br_2 on cyclooctatetraene in CH_2Cl_2 and $CHCl_3$ at 0-20°C.

$C_8H_8Br_4$ 2.5.7.8. Tetrabromobicyclo (0.2.4) octene 3.
Higher melting form: colourless prisms mp. 147-148°C. from benzene.
Lower melting form: prisms from ligroin mp. 94°C.
By action of 2 moles Br_2 in $CHCl_3$ on cyclooctatetraene.

$C_8H_8Br_6$ 2.3.4.5.7.8. Hexabromobicyclo (0.2.4) octane.
Colourless prisms from benzene mp. 153-154°C.
By action of 3 moles Br_2 in chloroform on cyclooctatetraene.

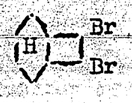
$C_8H_{10}O$ p phenylethylalcohol.
bp. /760 mms.
From styryl acetate hydrogenation and hydrolysis.
Phenylurethane mp. 80-81°C. p. nitrobenzoate mp. 60-61°C.

$C_8H_{10}Cl_2$ 7.8 Dichlorobicyclo (0.2.4) octene (2 or 3)
Colourless liquid bp. 104°C/13 mms. n D/20 1.5242.
By catalytic hydrogenation of $C_8H_8Cl_2$ in di-isopropyl ether in presence $Ni/CaCO_3$ catalyst.

$C_8H_{12}O_2$ Hexahydro.O. phthalaldehyde.
Colourless liquid bp. 91-94°C./3 mms.
By oxidation of glycol $C_8H_{14}O_2$ with $Pb(OAC)_4$ ~~polymerises very rapidly.~~

$C_8H_{12}Cl_2$ 7.8 dichlorobicyclo (0.2.4) octane
Colourless liquid bp. 104°C/12 mms. n D/20 1.5069. D 20/4 1.1887.
By catalytic hydrogenation of $C_8H_8Cl_2$ in presence of Pd or nickel catalyst.

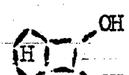
$C_8H_{12}Br_2$ 7.8 dibromobicyclo (0.2.4) octane.
Colourless liquid bp. 91°C/1.2 mms. n D/20 1.5583. D 20/4 1.7050
By catalytic hydrogenation of $C_8H_8Br_2$



$C_8H_{14}O$ Cycloheptylformaldehyde.
Colourless liquid bp. 61°C/8 mms.
From dimethyl ether $C_{10}H_{20}O_2$ by treatment with dilute sulphuric acid.
Semicarbazone mp. 155-156°C.

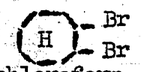
Cyclooctene oxide.
White crystals mp. 38-40°C bp. 88°C/25 mms.
By action of perbenzoic acid in chloroform on cyclooctene.

$C_8H_{14}O_2$ 7.8 Dihydroxybicyclo (0.2.4) octane.
Plates from benzene mp. 142°C.
From the diacetate $C_{12}H_{18}O_4$ by hydrolysis with methylalcoholic HCl.

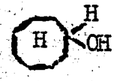


$C_8H_{14}O_4$ Suberic Acid.
mp. 138° - 140°C. HOOC CH_2 $[CH_2]_4$ CH_2.COOH
1) By oxidation of cyclooctene with HNO_3, CrO_3 or $KMnO_4$
2) " " " cyclooctane with HNO_3
3) " " " cyclooctanol with HNO_3

$C_8H_{14}Br_2$ 1.2. Dibromocyclooctane.
Colourless liquid bp. 123-124°C./5 mms.
By addition of 1 mole Br_2 to cyclooctene in chloroform.



$C_8H_{16}O$ Cyclooctanol.
bp. 74°C/3 mms. 100-101°C/15 n D/20 1.4871.
D 20/4 0.9740.
By catalytic hydrogenation of the oxide C_8H_8O

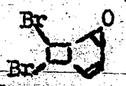


$C_8H_7O_2Cl$ Constitution unknown.
Needles from MeOH mp. 139-141°C.
By oxidation of $C_8H_8Cl_2$ with perbenzoic acid in $CHCl_3$, removal of $CHCl_3$, residue taken up in ether and extracted with NaOH solution.

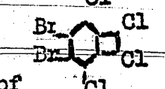
$C_8H_7O_2Br$ Constitution unknown.
Colourless needles from benzene mp. 144°C.
From $C_8H_8Br_2$ as above.

$C_8H_8OCl_2$ 7.8 Dichlorobicyclo (0.2.4) octene.2. oxide (4.5)
Colourless prisms from petrol ether mp. 72-75°C.
By action of perbenzoic acid in $CHCl_3$ on $C_8H_8Cl_2$

$C_8H_8O\ Br_2$ 7.8 Dibromobicyclo (0.2.4) octene. 2. oxide (4.5)
Colourless needles from ligroin mp. 88°C.
By action of perbenzoic acid in $CHCl_3$ on $C_8H_8Br_2$



$C_8H_8Cl_4Br_2$ 2.5.7.8. Tetrachloro 3.4 Dibromobicyclo. (0.2.4) octane.
Colourless prisms from MeOH mp. 100-101°C.
By action of Br_2 on tetrachloride $C_8H_8Cl_4$ of mp. 112°C in $CHCl_3$.



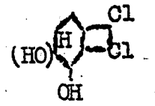
$C_8H_{10}O_2Cl_4$ Constitution unknown.
Colourless needles from Benzene mp. 140°C.
Oxidation of tetrachloride $C_8H_8Cl_4$ of mp. 112°C. with $KMnO_4$ in acetone.

$C_8H_{10}Cl_2Br_2$ 7.8 Dichloro 3.4 dibromobicyclo (0.2.4) octane
Colourless needles from CH_3OH mp. 124-125°C.
By action 1 mole Br_2 on $C_8H_{10}Cl_2$ in CH_2Cl_2

$C_8H_{10}O_4Cl_2$ Constitution unknown.
Colourless needles from benzene mp. 129-130°C.
A dibasic acid formed by the oxidation with HNO_3 of the hydrogenated oxide $C_8H_{10}OCl_2$ mp. 75°C.

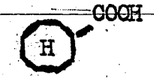
$C_8H_{10}O_5Cl_2$ Constitution unknown.
Needles from water mp. 218-220°C.
A dibasic acid produced by the oxidation of $C_8H_8Cl_4$ with $KMnO_4$ in acetone.

$C_8H_{12}OCl_2$ 2(or 3) hydroxy.7.8 dichlorobicyclo (0.2.4) octane.
mp. 80-81°C.
By catalytic hydrogenation of the oxide $C_8H_8OCl_2$ with Pd. catalyst.

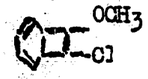


C_8H_8ONCl Constitution unknown.
Colourless powder mp. 87-88°C. (decomp.)
Addition product of NOCl and cyclooctatetraene.

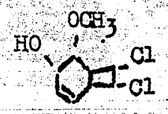
$C_9H_{16}O_2$ Cyclooctane carboxylic acid.
(bp. 135-138°C/3.3 mms.)
From cyclooctane by reaction with CO and H_2O in presence of $Ni(CO)_4$.



$C_9H_{11}OCl$ Probably 7. Methoxy 8 chloro bicyclo (0.2.4) octadiene (2.4)
Colourless liquid bp. 87-90°C/10 mms.
By action of NaOMe on the dichloride in the cold (5-10°C.)



$C_9H_{12}O_2Cl_2$ 4 Hydroxy-5 methoxy - 7.8 dichloro-bicyclo (0.2.4) octene 2.



The position of the HO and CH_3O can be interchanged.
Plates from ligroin mp. 80-81°C.
From the oxide $C_8H_8OCl_2$ by warming with methyl alcohol.

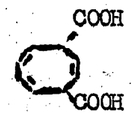
$C_{10}H_{14}$ 3.6 Endocyclobutylene cyclohexene -1.

Colourless oil bp. 80-82°C/30 mms.
From the phosphate of 3.6 endocyclobutylene-hexylamine -1 by distillation.

$C_{10}H_{16}$ 3.6 Endocyclobutylenecyclohexane.

bp. 100-120°C/15 mms. (air bath temperature)
By hydrogenation of $C_{10}H_{14}$ (above) in acetic acid in the presence of Pd - animal charcoal.

$C_{10}H_8O_4$ Cyclooctatriene (1.3.6) dicarboxylic acid (5.8)



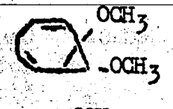
1) Yellow acid mp. 240-260°C.
2) White acid mp. 150°C.
By action of CO_2 on the lithium compound of cyclo-octatetraene.

$C_{10}H_{10}O_2$ Styryl acetate. $C_6H_5.CH = CH.O.CO.CH_3$
bp. 80°C/0.5 mms. n D/20 1.5513.
By action of acetic acid on the dichloride $C_8H_8Cl_2$

$C_{10}H_{12}O_2$ β phenylethylacetate. $C_6H_5.CH_2.CH_2.O.CO.CH_3$

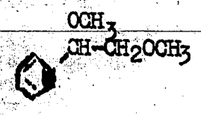
1) By catalytic hydrogenation of styryl acetate.
2) By action of Zn dust and acetic acid on $C_8H_8Cl_2$

$C_{10}H_{14}O_2$ 7.8 dimethoxybicyclo (0.1.5) octadiene 2.5



Almost colourless liquid bp. 79°C/8 mms.
n D/20 1.5088. D 20/4 1.0206
By action of NaOMe on $C_8H_8Cl_2$

$C_{10}H_{14}O_2$ Phenylglycol dimethyl ether.

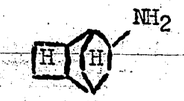


Colourless liquid bp. 93°/10 mms.
D 20/4 1.0047. n D/20 1.4946. From the dichloride $C_8H_8Cl_2$ and methanol.

$C_{10}H_{16}O$ Probably 3.6 Endocyclobutylene 3. cyclohexanol 1.

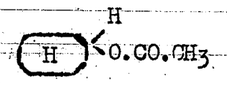
White crystals from ligroin mp. 138-140°C.
From amine (below) with HNO_2. ($NaNO_2$ & H_2SO_4)

$C_{10}H_{17}N$ 3.6 Endocyclobutylenecyclohexylamine.



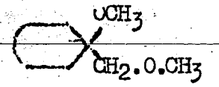
bp. 110-111°C./16 mms.
By the decomposition of the acid azide of the corresponding carboxylic acid.

$C_{10}H_{18}O$ Cyclooctanol acetate.



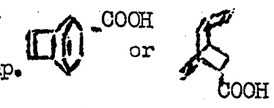
Colourless liquid bp. 75-76°C/3 mms.
From cyclooctene and acetic and sulphuric acids.

$C_{10}H_{20}O_2$ 1 Methoxy. 1 methoxy methyl cycloheptane.



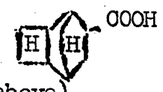
Colourless liquid bp. 88°C/13 mms.
74-75°/8 n D/20 1.4546. D 20/4 0.9494
By catalytic hydrogenation of the ether $C_{10}H_{14}O_2$ (from $C_8H_8Cl_2$ and NaOMe) with Pd or Ni catalyst.

$C_{11}H_{12}O_2$ 3.6 Endocyclobutadienylenecyclo-hexene 4 carboxylic acid 2.



Colourless crystals from water mp. 112-113°C.
By condensation of cyclooctatetraene with acrylic acid at 150°C.

$C_{11}H_{16}O_2$ 3.6 Endocyclobutylene cyclohexane carboxylic acid 2.

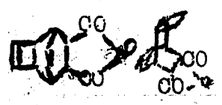


Needles from water mp. 85-86°C.
By catalytic hydrogenation of $C_{11}H_{12}O_2$ (above) with Pd or Nickel catalyst.

$C_{12}H_8O_3$ 3.6 Endocyclobutadienylenecyclo-hexadiene 1.4 dicarboxylic acid 1.2 anhydride.

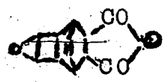
Anhydride (by means Ac_2O) of the acid produced by hydrolysis of the addition product of cyclooctatetraene and acetylene dicarboxylic ester.

$C_{12}H_{10}O_3$ 3.6 Endocyclobutadienylenecyclohexen.4. cis dicarboxylic acid 1.2 anhydride.



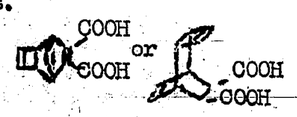
White crystals from monochlorobenzene mp. 167°C.
From cyclooctatetraene and maleic anhydride.

$C_{12}H_{10}O_4$ Addition product from Cyclooctatriene (1.3.5) 7.8 oxide and maleic anhydride.



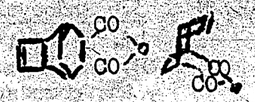
Needles from monochlorobenzene mp. 205-206°C.
Diels Alder reaction of constituents.

3.6 Endocyclobutadienylenecyclo-hexadiene 1.4 dicarboxylic acid.



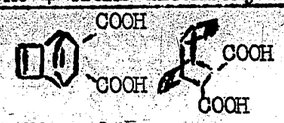
White powder mp. 158-160°C.
By hydrolysis of anhydride (above)

$C_{12}H_{12}O_3$ 3.6 Endocyclobutadienylenecyclohexane cis dicarboxylic acid anhydride.



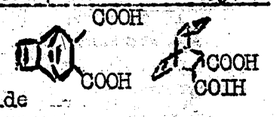
White crystals from benzene
mp. 140-142°C.
By catalytic hydrogenation or maleic anhydride addition product in alkaline solution in presence of Pd. catalyst and transformation to anhydride by distillation.

$C_{12}H_{12}O_4$ 3.6 Endocyclobutadienylenecyclohexene 4 trans dicarboxylic acid 1.2



White crystals from methanol
mp. 218°C.
From heating the dimethyl ester of the cis acid (see below) with NaOMe and hydrolysing the ester of the trans acid so formed.

3.6 Endocyclobutadienylenecyclohexene 4 cis dicarboxylic acid 1.2



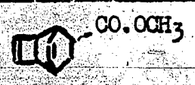
White needles from methanol/water
mp. 167°C (decomp.)
By action of dilute NaOH on anhydride

$C_{12}H_{10}O_3$

Probably 3.6 Endocyclobutylene 3.4' oxidocyclohexane dicarboxylic acid 1.2 anhydride.

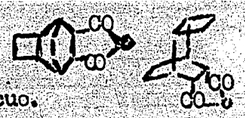
Colourless prisms from benzene
mp. 140-145°C.
By catalytic hydrogenation of the addition compound of cyclooctatriene (1.3.5) 7.8 oxide and maleic anhydride.

$C_{12}H_{14}O_2$ 3.6 Endocyclobutadienylenecyclohexene 4 carboxylic acid 1. methyl ester.



bp. 135° - 140°C/12 mms (air bath temp)
By esterification of the addition product of cyclooctatetraene and acrylic acid.

$C_{12}H_{14}O_3$ 3.6 Endocyclobutylenecyclohexane cis dicarboxylic acid 1.2 anhydride.



Colourless crystals mp. 130-132°C
bp. 198°C/10 mms.
By distillation of free acid in vacuo.

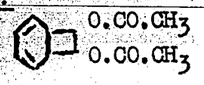
Probably "ditto" (Exo compound)

Colourless crystals from cyclohexane
mp. 154-155°C.
By catalytic hydrogenation of 3.6 Endocyclobutadienylene-cyclohexadiene 1.4 di carboxylic acid 1.2 anhydride in acetic acid in the presence of Pd. charcoal.

$C_{12}H_{14}O_4$ 3.6 Endocyclobutadienylenecyclohexene cis dicarboxylic acid 1.2.

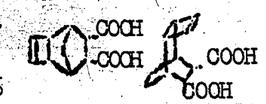
Crystals from benzene mp. 149°C. (decomp.)
By catalytic hydrogenation of the addition compound of cyclooctatetraene and maleic anhydride in alkaline solution in presence of Pd.

7.8 Diacetoxybicyclo (0.2.4) octadiene 2.4.



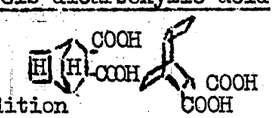
Colourless needles from ligroin mp. 66°C.
From the dichloride $C_8H_8Cl_2$ by action of KAC in glacial acetic acid.

3.6 Endocyclobutadienylenecyclohexane trans dicarboxylic acid 1.2



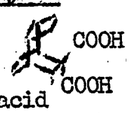
Colourless crystals from methanol/water mp. 212-213°C.
1) By partial hydrogenation of 3.6 endocyclobutadienylenecyclohexene 4 trans dicarboxylic acid 1.
2) By heating the dimethyl ester of the cis acid with NaOMe and hydrolysing the trans ester.

$C_{12}H_{16}O_4$ 3.6 Endocyclobutylenecyclohexane cis dicarboxylic acid 1.2



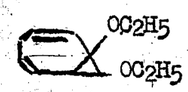
Crystals from ether or methanol/water mp. 172°C (decomp.)
By catalytic hydrogenation of addition product of cyclooctatetraene and maleic anhydride in alkaline solution in the presence of Pd or Ni catalyst.

3.6 Endocyclobutylenecyclohexane trans dicarboxylic acid 1.2



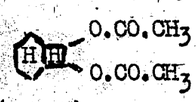
White powder mp. 228-229°C.
By catalytic hydrogenation of 3.6 Endocyclo-butadienylenecyclohexane trans dicarboxylic acid 1.2 or by action of NaOMe on dimethyl ester of the cis acid (above) followed by hydrolysis of the trans ester formed.

$C_{12}H_{16}O_4$ 7.8 Diethoxy 0.1.5. bicycloocta 2.5 diene.



Colourless liquid bp. 101-102°/2 mm.
from the dibromide $C_8H_8Br_2$ and sodium ethoxide.

$C_{12}H_{18}O_4$ 7.8 Diacetoxy bicyclo (0.2.4) octane.



Colourless liquid bp. 105°C/0.5 mms.
$n_D/20$ 1.4662. D 20/4 1.1025.
By hydrogenation of 7.8 Diacetoxybicyclo (0.2.4) octadiene 2.4.

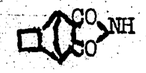
$C_{12}H_{10}O_4Br_2$ 3.6 Endo 3'4' dibromocyclobutylenecyclohexadiene 4 dicarboxylic acid 1.2.

Crystals from acetic acid/water mp. 205-207°C.
By hydrolysis of addition compound of $C_8H_8Br_2$ and acetylene dicarboxylic ester.

$C_{12}H_{11}O_2N$ 3.6 Endocyclobutadienylenecyclohexene 4 dicarboxylic acid 1.2 imide.

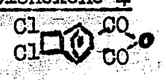
mp. 252-253°C (with sublimation)
By action of aqueous ammonia at 120°C on addition product of cyclooctatetraene and maleic anhydride.

$C_{12}H_{15}O_2N$ 3.6 Endocyclobutylenecyclohexane dicarboxylic acid 1.2 imide.



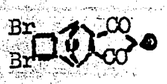
Colourless crystals mp. 212-213°C.
By action of aqueous ammonia at 120°C on the anhydride.

$C_{12}H_{10}O_3Cl_2$ 3.6 Endo 3'4' dichlorocyclobutylene cyclohexene 4 dicarboxylic acid 1.2 anhydride.



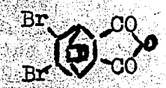
Higher melting form: plates from monochlorobenzene 267-268°C.
Lower melting form: needles from benzene mp. 195-196°C.
Both forms obtained from the dichloride $C_8H_8Cl_2$ with maleic anhydride.

$C_{12}H_{10}O_3Br_2$ Dibromide corresponding to above.



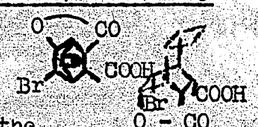
Prisms from monochlorobenzene mp. 205°C.
From dibromide $C_8H_8Br_2$ and maleic anhydride.

3.6 Endocyclobutadienylene 4.5 dibromocyclohexane dicarboxylic acid 1.2 anhydride.



White crystals from acetic acid.
By hydrolysis of the dimethyl ester of free acid with conc. H_2SO_4.

$C_{12}H_{11}O_4Br$ 3.6 Endocyclobutadienylenecyclohexane 4 bromo. 1.3 lactone. 2. carboxylic acid.



Colourless crystals from methyl alcohol/water mp. 238°C (with decomposition).
By action of 1 mol Br_2 in MeOH on the potassium salt of 3.6 Endocyclobutadienylene cyclohexene dicarboxylic acid 1.2.

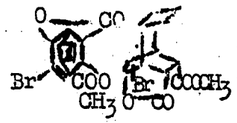
$C_{12}H_{12}O_4Br_2$ 3.6 Endocyclobutadienylene 4.5 dibromocyclohexane dicarboxylic acid.

Rosettes from methanol/water mp. 234-236°C.
By hydrolysis of the Dimethyl ester with methanolic KOH.

$C_{13}H_{18}O_4$ 3.6 Endocyclobutylene cyclohexane cis dicarboxylic acid 1.2 monomethyl ester.

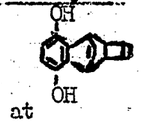
Needles from benzene and ligroin mp. 95-96°C.
By treatment of the anhydride of the acid with methanol at 120°C.

$C_{13}H_{13}O_4Br$ 3.6 Endocyclobutadienylene 4 bromocyclo hexane 1.5 lactone 2 carboxylic acid methyl ester.



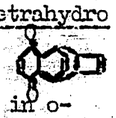
mp. 177-178° from chloroform/ligroin
By treating 3.6 Endocyclobutadienylene cyclohexane 1.2 dicarboxylic acid dimethyl ester with 1 mol bromine in methanol.

$C_{14}H_{12}O_2$ 1.4 Dihydroxy 4.8 cyclobutadienylene. 5.8 dihydronaphthalene.



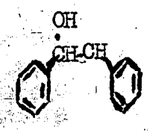
White crystalline solid mp. 133° from benzene or water. By treatment of cyclooctatetraene with benzoquinone at 180-200° or by heating the quinone adduct of mp. 141° to 180-200°.

5.8 Endocyclobutadienylene. 5.8.9.10. tetrahydro 1.4 naphthoquinone.



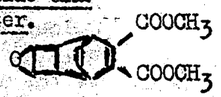
Yellow crystals mp. 141° from alcohol.
From cyclooctatetraene and benzoquinone in o-dichlorbenzene at 140°.

$C_{14}H_{14}O$ Phenyl benzyl carbinol.



mp. 68-71° from ligroin. From phenyl acetaldehyde and phenylmagnesium bromide.
Used for identification of phenyl-acetaldehyde.

$C_{14}H_{14}O_5$ Adduct from 1.3.5 Cyclooctatriene 7.8 oxide and acetylene dicarboxylic acid dimethyl ester.



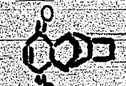
Colourless crystals mp. 103° from acetylene dicarboxylic dimethyl ester and 1.3.5 cyclooctatriene 7.8 oxide.

$C_{14}H_{16}O_2$ 1.4 Dihydroxy 5.8 endocyclobutylene 5.6.7.8. tetrahydronaphthalene.

White crystals mp. 178-180° from benzene. By hydrogenation of the quinone adduct of mp. 133° over palladium.

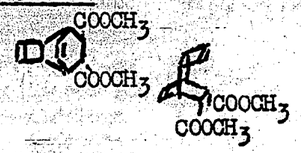
$C_{14}H_{16}O_2$ 5.8 Endocyclobutylene 5.6.7.8.9.10. hexahydro naphthaquinone. (apparently).

White crystals mp. 251-252° from monochlorbenzene. Produced in small quantities on hydrogenation of the quinone adduct $C_{14}H_{12}O_2$, of mp. 141° in suspension in methanol.



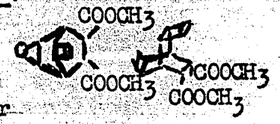
$C_{14}H_{16}O_4$ 3.6 Endocyclobutadienylene cyclohex-4-5-ene. 1.2 dicarboxylic acid dimethyl ester.

Colourless oil bp. 160°/1 mm. by esterification of the acid using methyl alcoholic sulphuric acid.



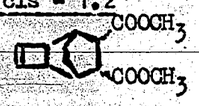
$C_{14}H_{16}O_5$ 3.6 Endocyclobutadienyl 4.5 oxydo- cyclohexane cis 1.2 dicarboxylic acid dimethyl ester.

Crystalline from cyclohexane. mp. 85-6°. From 3.6 endocyclobutadienylene cyclo-hex-4.5-ene 1.2 dicarboxylic acid dimethyl ester by perbenzoic acid in chloroform.



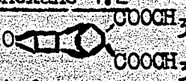
$C_{14}H_{18}O_4$ 3.6 Endocyclobutadienylene cyclo-hexane cis - 1.2 dicarboxylic acid dimethyl ester.

Colourless crystals mp. 25-30°, bp. 158-160°/2 mm. From the anhydride by treatment with methyl alcoholic sulphuric acid.



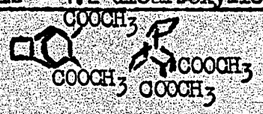
$C_{14}H_{18}O_5$ 3.5 Endo, 3'.4' oxydo cyclobutylene cyclohexane 1.2 dicarboxylic acid dimethyl ester.

Colourless needles mp. 86-87° from ether. From catalytic hydrogenation of the adduct from cyclooctatriene oxide and acetylene dicarboxylic dimethyl ester.



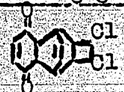
$C_{14}H_{20}O_4$ 3.5 Endocyclobutylene cyclohexane cis - 1.2 dicarboxylic acid dimethyl ester.

Colourless rosettes mp. 62-63° bp. 160°/4 mm. from esterification of the anhydride with methyl alcoholic sulphuric acid.



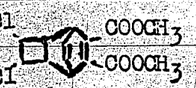
$C_{14}H_{12}O_2Cl_2$ Adduct of benzaquinone and the dichloride $C_8H_8Cl_2$

Needles mp. 174° from benzene. From condensation of $C_8H_8Cl_2$ and benzaquinone in benzene.



$C_{14}H_{14}O_4Cl_2$ Adduct from dimethyl acetylene dicarboxylate and $C_8H_8Cl_2$.

Colourless plates mp. 125-126° from methanol. From condensation of the dichloride $C_8H_8Cl_2$ and dimethyl acetylene dicarboxylate in benzene.



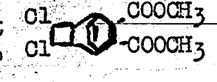
$C_{14}H_{14}O_4Br_2$ Adduct from the dibromide $C_8H_8Br_2$ and dimethyl acetylene dicarboxylate.

Leaflets mp. 134-135 from methanol. From condensation of the dibromide with dimethyl acetylene dicarboxylate in benzene.

$C_{14}H_{16}O_4Cl_2$ Adduct from the dichloride $C_8H_8Cl_2$ and dimethyl maleate.

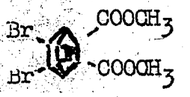
1) Leaflets mp. 126-127° from methanol from esterification of the dichloride/maleic anhydride adduct of mp. 195-196° with methyl alcohol/sulphuric acid.

2) Needles mp. 220° from methanol from esterification of the dichloride/maleic anhydride adduct of mp. 268° with methyl alcohol/sulphuric acid.



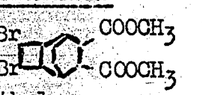
$C_{14}H_{16}O_4Br_2$ 3.5 Endocyclobutadienylene 4.5 dibron cyclohexane 1.2 dicarboxylic acid dimethyl ester.

Crystals mp. 225-227° from chloroform/ligroin. From 3.5 Endocyclobutadienylene 4.5 cyclohexene 1.2 dicarboxylic acid dimethyl ester.



$C_{14}H_{18}O_4Br_2$ 3.5 Endo 3'.4' dibromocyclobutylene cyclohexane 1.2 dicarboxylic acid dimethyl ester.

Crystals mp. 148° from methanol From catalytic hydrogenation of the adduct from the dibromide and dimethyl acetylene dicarboxylate.



$C_{15}H_{12}O_2$ Styryl benzoate.

Colourless oil bp. 150°/0.1 mm. $n_D/20°$ 1.6087. From the dichloride $C_8H_8Cl_2$ and perbenzoic acid in chlorbenzene.

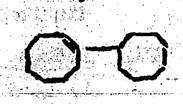
$C_{16}H_{16}$ **Dimeric Cyclooctatetraene (liquid)**
Colourless oil solidifying below 0°C to a white glass. bp. 136/1 mm. D 20/4 1.1044. n D/20 1.5868. From heating cyclooctatetraene in an atmosphere of nitrogen.

Dimeric cyclooctatetraene (crystalline).
Colourless crystals mp. 43-44° from ether. Bp.138°/1 mm. Produced along with the liquid dimer on heating cyclooctatetrene in absence of air.

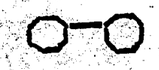
$C_{16}H_{20}$ **Hydrocarbon from hydrogenation of $C_{16}H_{16}$ (liquid dimer)**
Colourless crystalline mass. mp. 30°, bp. 136°/1 mm. From catalytic hydrogenation of the liquid dimer $C_{16}H_{16}$.

$C_{16}H_{22}$ **Hydrocarbon from hydrogenation of the dimer of mp. 43-44°C.**
Colourless oil bp. 127-128°/0.5 mm. D 20/4 1.1364 n D/20 1.5668. From hydrogenation of the dimer of mp. 43-44° over a nickel or palladium catalyst.

$C_{16}H_{23}$ **Cyclooctenyl cyclooctane.**
Colourless oil bp. 130-131°/1 mm. D 20/4 0.9221. n D/20 1.4994.



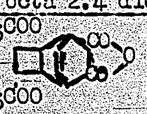
$C_{16}H_{30}$ **Cyclooctyl cyclooctane.**
Colourless oil bp. 135-140°/1 mm. From hydrogenation of cyclooctenyl cyclooctane.



$C_{16}H_{14}Cl_2$ **Constitution unknown.**
Colourless crystals mp. 249-250°, produced in small amount on treating the dichloride $C_8H_8Cl_2$ with zinc dust & methyl alcohol or acetic acid.

$C_{16}H_{16}O_2$ **Dimethyl ether of the quinone adduct of mp.133°**
Crystals mp. 106° from methanol. From methylation of the quinone adduct of mp. 133° with dimethyl sulphate and potassium hydroxide solution.

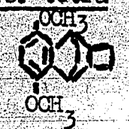
$C_{16}H_{16}O_7$ **Adduct from 7.8, diacetoxy 0.2.4 bicyclo octa 2.4 diene, and maleic anhydride.**
Colourless prisms mp. 131° from benzene/ligroin.



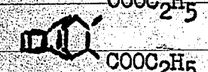
$C_{16}H_{16}Cl_4$ **Dimeric dichloride.**
Prisms mp. 190° from acetic acid. On long standing or more rapidly on warming reforms the dichloride $C_8H_8Cl_2$.

$C_{16}H_{16}Br_4$ **Dimeric dibromide.**
Colourless prisms mp. 218° from chlor-benzene. On long standing or more rapidly on warming reforms the dibromide $C_8H_8Br_2$.

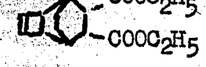
$C_{16}H_{20}O_2$ **1.4 Dimethoxy, 5.8 endocyclobutylene 5.6.7.8. tetra hydronaphthalene.**
Crystals mp. 119-120° from ethanol. From hydrogenation of the dimethyl ether $C_{16}H_{16}O_2$.



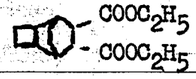
$C_{16}H_{20}O_4$ **3.6 Endocyclobutadienylene cyclohex-4-ene cis 1.2 dicarboxylic acid diethyl ester.**
mp. 57-58° Bp. 156-8/1 mm. From esterification of the maleic anhydride adduct with ethyl alcohol/sulphuric acid.



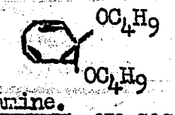
$C_{16}H_{22}O_4$ **3.6 Endocyclobutadienylene cyclohexane 1.2 cis dicarboxylic acid ethyl ester.**
Colourless oil bp. 164-167°/2 mm. from the acid anhydride by esterification with ethyl alcohol/sulphuric acid.



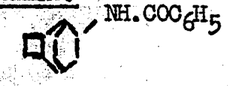
$C_{16}H_{24}O_4$ **3.6 Endocyclobutylene cyclohexane 1.2. dicarboxylic acid diethyl ester.**
Colourless liquid bp. 177-178/3 mm. From the anhydride by esterification with ethyl alcohol/sulphuric acid.



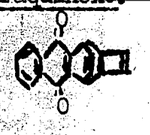
$C_{16}H_{26}O$ **7.8 Dibutoxy 0.1.5. bicycloocta -3.5 - diene.**
Colourless liquid bp. 126-127/2 mm. From the dibromide $C_8H_8Br_2$ and sodium butoxide.



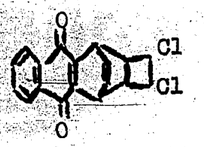
$C_{17}H_{21}ON$ **3.6 Endocyclobutylene .1. cyclohexylbenzylamine.**
Crystalline. Mp. 184° from the amine by benzoylation using benzoylchloride in pyridine.



$C_{18}H_{12}O_2$ **1.4 Endocyclobutadienylene 1.4 dihydroanthraquinone.**
Golden crystals mp. 192° from butanol. From condensation of cyclooctatetrene and anthraquinone followed by atmospheric oxidation in alkaline solution.

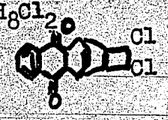


$C_{18}H_{12}O_2Cl_2$ **1.4 Endocyclo. 3.4 dichlorocyclobutylene 1.4 dihydroanthraquinone.**
Bright yellow needles mp. 170-180° from benzene. From the naphthoquinone adduct of the dichloride by air oxidation in alkaline solution.



$C_{18}H_{14}O_2Cl_2$ **Naphthoquinone adduct of the dichloride $C_8H_8Cl_2$**

Colourless needles mp. 221° from benzene, by heating the dichloride with naphthoquinone in benzene.

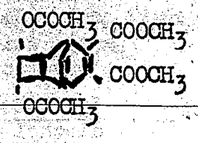


$C_{18}H_{16}O_4$ **Diacetate of the benzoquinone/cyclooctatetraene adduct of mp. 133°**

Needles mp. 137-138° from methanol By treatment of the benzoquinone/cyclooctatetraene adduct with acetic anhydride.

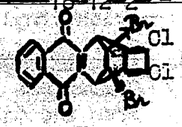
$C_{18}H_{20}O_8$ **Adduct from 7.8 diacetoxy. 0.2.4. octa 2.4 diene and dimethylacetylene dicarboxylate.**

Colourless prisms mp. 84° from benzene/ligroin. From condensation of the diacetate $C_{12}H_{14}O_4$ in the dimethylacetylene dicarboxylate.



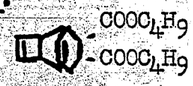
$C_{18}H_{12}O_2Cl_2Br_2$ **Dibromide of the naphthaquinone adduct $C_{18}H_{12}O_2Cl_2$**

Prisms mp. 300° from chlorobenzene. Obtained by treating the adduct $C_{18}H_{12}O_2Cl_2$ with 1 mol bromine in chloroform.



$C_{20}H_{28}O_4$ **3.6 Endocyclobutadienylene cyclohex-4-ene 1.2 dicarboxylic acid di-n-butyl ester.**

Bp. 178-179°/1 mm. mp. 35-38° From the corresponding anhydride by heating with butanol and sulphuric acid in presence of xylene.



3.6 Endocyclobutadienylene cyclohex-4-ene 1.2 dicarboxylic acid di-iso-butyl ester.

Bp. 178°/1 mm. mp. 52-55°. From the anhydride and isobutanol and sulphuric acid in xylene.

$C_{20}H_{30}O_4$ **3.6 Endocyclobutadienylene cyclohexane 1.2 dicarboxylic acid dibutyl ester.**

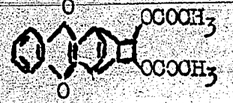
Colourless oil bp. 188-190°/2 mm. From the anhydride by heating with butanol and sulphuric acid in benzene.

$C_{20}H_{32}O_4$ **3.6 Endocyclobutylene cyclohexane 1.2 dicarboxylic acid dibutyl ester.**

Colourless oil bp. 210°/3 mm. From the anhydride by heating with butanol and sulphuric acid in benzene.

$C_{22}H_{18}O_6$ **Dehydrogenation product of the naphthoquinone adduct of the diacetate $C_{12}H_{14}O_4$**

Bright yellow leaflets mp. 190-200° from dilute methanol. By air oxidation of the adduct $C_{22}H_{20}O_6$ in alkaline solution.



$C_{22}H_{28}O_4$ **Substances of unknown constitution.**

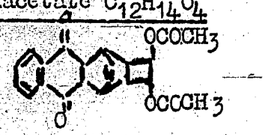
1) Viscous oil bp. 200-205/0.5 mm. From treatment of the hydrocarbon $C_{16}H_{16}$ mp. 43-44° with 2 mols carbon monoxide and 2 mol water in the presence of nickel carbonyl and esterification of the acid produced with methyl alcohol/sulphuric acid.

2) Viscous oil bp. 208-218°/0.8 mm. From the liquid hydrocarbon $C_{16}H_{16}$ by treatment with carbon monoxide, 2 mol, and water 2 mol, in the presence of nickel carbonyl and esterification of the acid produced with methyl alcohol/sulphuric acid.

3) Viscous oil bp. 202-204°/0.6 mm. From cyclooctatetraene by the action of carbon monoxide 1 mol and water 1 mol. There is simultaneous dimerisation and the acid produced is esterified with methyl alcohol/sulphuric acid.

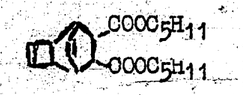
$C_{22}H_{20}O_6$ **Adduct from naphthoquinone and the diacetate $C_{12}H_{14}O_4$**

Prisms mp. 160-161° from methanol. From condensation of 7.8 diacetoxy 0.2.4. bicyclo-octa-2.4-diene with naphthoquinone.



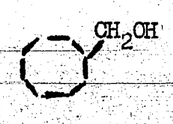
$C_{22}H_{34}O_4$ **3.6 Endocyclobutadienylene cyclohex-4-ene 1.2 dicarboxylic acid diamyl ester.**

Crystalline mp. 58-60° Bp. 198-200/2 mm. From the maleic anhydride adduct of cyclooctatetraene by heating with amyl alcohol and sulphuric acid in benzene.



$C_9H_{12}O$ **Cyclooctyl carbinol.**

Colourless liquid bp. 106°/7 mm. From cyclooctatetraene or cyclooctene by the action of water gas in the presence of a cobalt catalyst.

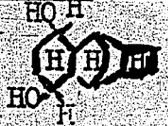


$C_{14}H_{18}O_2$ 1.4 Endocyclobutylene 5.8 diketodecaline.

Colourless crystals mp. 131-133°. From hydrogenation of the quinone adduct of mp. 141° with a palladium catalyst in solution in tetrahydrofuran.

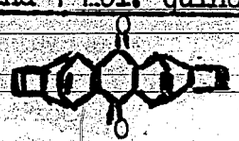
$C_{14}H_{22}O_2$ 1.4 Endocyclobutylene 5.8 dihydroxy decaline.

Crystalline mp. 181° From hydrogenation of the quinone adduct of mp. 141° (under pressure) using a nickel catalyst.



$C_{22}H_{20}O_2$ Adduct from 2 mols cyclooctatetrene and 1 mol. quinone.

Yellow crystal mp. 223° as a by-product from the condensation of cyclooctatetrene with quinone.



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FINAL REPORT No. 247.
ITEM No. 22.

GERMAN CHEMICAL PLANT WITH PARTICULAR REFERENCE TO CENTRIFUGES

Broadbent, J.

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REC'D JUL 1946
TIC A.P.I.U.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON - H.M. STATIONERY OFFICE.

GERMAN CHEMICAL PLANT

WITH PARTICULAR REFERENCE TO CENTRIFUGES

Reported By

F. BROADBENT, B.Sc. A.M.I. Chem.E.

BIOS Target Numbers
C22/2602, C22/2569, C22/2191, 22/1(c)
22/1(m), C22/1657, C22/2192, C22/2190, C22/2603

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

32, Bryanston Square, W.1.

(1946)

21 p.

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REPORT NO. 1.

ELMORE METALL A.G. C22/2602

SCHLADERN ON SIEG.

Object of Visit To investigate the manufacture of electrolytically deposited copper shells.

Person Interviewed Herr Weber (Manager).

Condition of Works The factory is in good working order.

Damaged bridge across river needs repair.

Ownership This is a British owned factory with a capital of approximately £100,000. The Head Office pre-war was Algoa House, 41, Moorfields, London E.C.2. Directors pre-war were:-

1. Oscar Becker (Chairman) located at Branders, Goldschmidt & Co., also at "Verosa", Sandy Drive, Nr. Cobham, Surrey; 9-13 King William Street, London, E.C.4.

2. C. A. Jenkins (Sales Director) 132, Woodlands Avenue, Wanstead, London.

3. Hugh Nettlefold, 163, Euston Road, London, N.W.1.

4. R. T. Edge, Stone Quarry House, Hammer Vale, Haslemere, Surrey.

Present Control The Manager in charge is Herr Weber, who has been employed by the firm for twenty-five years. He was working under Nazi control during the war,

Present Control (Contd.)

and is now Caretaker Manager by consent of the Military Government at Siegburg. He is seventy-one years old and ready to retire.

Technical Data

These works are divided into two sections, a tube drawing section for copper steel and zinc with old machinery of little interest; and a section making electrolytically deposited copper cylinders up to 100" diameter, of high purity and excellent mechanical properties.

Electrical Supply

The factory is situated on the river Sieg and has its own generating plant driven from water turbines supplied from the river, with an average output of 100,000 k.W.H. per month. In addition they take power from the grid using approximately the same amount as they generate themselves. The bulk of the grid supply is taken in summer when the river is low.

Details Regarding Electrolytically Deposited Copper Cylinders

These are produced in lead lined wooden tanks. The cylinder is deposited on a wooden former coated with a conductor. The finish is obtained by a small stone block which presses against the cylinder of copper as it is deposited. The cylinder rotates and the stone block traverses very slowly backwards and forwards over the surface of the cylinder as it is being deposited. The rate of deposition is approximately 1/8" per week of 168 hours.

Recommendations

1. That steps be taken to establish contact between the British Directors of this company and Herr Weber at their works.

2. That the electrolytically deposited copper cylinder section of the plant be started up and its production allocated to British Industry.

Recommendation 2. (Contd.)

Manufacturers of Calico Printing Machinery, Paper Making Machinery, and Centrifuges use these cylinders.

These recommendations were included in the Interim Report made to B.A.O.R. Head Quarters on October 27th, 1945.

REPORT NO. 2.

H. KRANTZ MASCHINENFABRIK C22/2569

AACHEN

Object of Visit To determine whether there has been any new developments by these people in the manufacture of Hydro Extractors.

Persons Interviewed Herr Krantz (Director)

Condition of Works Badly damaged. The greater part of the plant has been destroyed.

Control This is an old established family business controlled by Herr Krantz.

Production This firm specializes in Hydro Extractors, confined to types suitable for laundries and textile works.

Technical Data The machines built are of sizes from 800 mm. to 1,800 mm. basket diameter. Speeds are lower than customary in Britain - for example - the standard speed of a Krantz Hydro Extractor with a 1,220 mm. diameter basket is 710 R.P.M.

The majority of the baskets made are of copper with gas welded seam. This firm is not progressive, and its designs are many years behind those available in Britain.

Recommendations That a British controlled firm takes over the markets filled pre-war by Krantz; and that as a first step records of machines supplied by Krantz be made available; and inquiries for new machines to Krantz be passed forward to a British firm.

REPORT NO. 3.

O. DÖRRIES A.G. - WORM BANNING & SEYBOLD

DÜREN C22/2191

Object of Visit — To find what developments have been made in the design of Erkensators - a special type of centrifugal machine for the removal of impurities from paper stock before it passes to the paper making machine.

Persons Interviewed — J. Lenzen (Sales and Commercial)

J. Braun (Engineer)

Condition of Works — Roof partly destroyed, but no crippling damage.

Production — This firm specializes in paper making machinery, including centrifugal grit extractors for the treatment of paper stock.

Technical Data — The largest Erkensator built pre-war was the number 4. machine, which is well known in Britain.

A number 4L. Erkensator has been developed which differs from the number 4. machine in the following respects.

1. Basket lengthened.

2. Speed increased from 570 R.P.M. to 720 R.P.M.

3. H.P. increased from 15-34.

Technical Data (Contd.)

4. The claim made regarding output is that this is increased from 60 c.m. per hour to between 75 and 80 c.m. per hour.

The following details were also of interest:-

(a) Herr Braun (Engineer) stated that when speeds higher than 720 R.P.M. were tried experimentally, too great an extraction of Kaolin was obtained in the Bolster.

(b) In view of the war-time shortage of copper a synthetic covering rather like vulcanite was used for machine parts coming into contact with the paper stock.

(c) Herr Lenzen (Sales and Commercial) stated that whilst this firm has built some hundreds of beaters for the German Nitrocellulose Industry, they have built no Erkensators for this purpose. This differs from war time practice in Britain.

Recommendations

That a British controlled firm takes over the market filled pre-war by the supply of Erkensators; and that records of machines supplied, and inquiries for new machines to O. Dorries be passed forward to a British firm.

REPORT NO. 4.

I. G. FARBENINDUSTRIE 22/1(c)

DORMAGEN

Object of Visit To obtain information regarding the continuously operating centrifuges installed in the Cellulose Acetate Plant.

Persons Interviewed Dr. Hofmann (Technical Director)

Dr. Ziegler (Engineer)

Technical Data The following details were obtained regarding these centrifuges, which were not actually in operation, since this section of the plant was shut down.

1. Maker of Centrifuges. Escher Wyss of Ravensburg.
2. Type. Horizontal, oscillating piston; wedge wire screen in basket.
3. Number Installed. 4.
4. Date of Installation. First unit about ten years ago. Latest unit 1940.
5. Basket Dimensions. Approximately 1,200 mm. diameter x 500 mm. long.
6. Piston Travel. Approximately 60 mm. 6 strokes per minute (variable)
7. Speed. 750 R.P.M.

Technical Data (Contd.)

8. Rate of Feed. To give 8 tons dry weight per 24 hours.

9. Solids in Feed. About 10% (variable)

10. Moisture content of discharged solids. 60 to 65%.

11. Constructional Materials. Generally Krupps V2A., for metal coming into contact with solids.

12. Motors. 25 k.W. main motor. 11 k.W. pump motor.

13. Mounting. These machines are all mounted on a heavy suspended floor. There is severe vibration if they are overfed.

Recommendations

That one of the Escher Eyss Continuous Centrifuges supplied in 1940 to the Cellulose Acetate Plant be forwarded to this country for stripping and examination.

REPORT NO. 5.

I. G. FARBENINDUSTRIE 22/1(m)

ELBERFELD

Object of Visit — To obtain information regarding the continuously operating centrifuges installed in the Hexamethylenetetramine Plant.

Persons Interviewed — Mr. Gardner (Military Government Officer in Charge)

Technical Data — The following details were obtained regarding the continuously operating centrifuges, which were not running, since this section of the plant was shut down.

1. Maker. Escher Wyss of Ravensburg.
2. Type. Horizontal, oscillating piston; wedge wire screen in basket.
3. Number Installed. 2.
4. Basket. Approximately $27\frac{1}{2}$" diam. x 14" long.
5. Piston Travel. Approx. 40 strokes per minute.
6. Speed. Approximately 900 R.P.M.
7. Main Motor. 11 k.W.
8. Output. Approximately 1 ton per hour.
9. Moisture content of discharged solids. approximately 5%.

Recommendations

That one of the Escher Wyss Continuous Centrifuges be forwarded to this country for stripping and examination.

REPORT NO. 6.

GLANSTOFF COURTALD C22/1657

BETWEEN COLOGNE AND DORMAGEN

Object of Visit — To look into the method used for the extraction of water from viscose cakes in the cake washing plant.

Persons Interviewed — Herr Fremery (Chemical Engineer)

Technical Data — This section of the plant was not in operation.

Two methods of extracting water from viscose cakes are used.

1. By multiple hinged candle hydro extractors.

2. By converted spinning pots.

Method 1. has the disadvantage of cake distortion, and is used for the heavier deniers.

Method 2. results in less distortion, but there is more handling than with method 1. Method 2. is used for lighter deniers.

Neither method is new. The machines used are similar in general design to those employed in Britain, but inferior in detail design, and of less suitable constructional design. The machine parts were badly corroded.

REPORT NO. 7.

MATTHES AND WEBER C22/2192

DUISBERG

Object of Visit — To inspect the centrifuges on the bi-carbonate plant.

Person Interviewed — Dr. Pohl (Director)

Technical Data — The plant was not running. The following centrifuges are installed.

1. On crude bi-carbonate.

Maker. Escher Wyss.

Type. Horizontal spindle with full width plough.

Number Installed. 3.

Basket. Approximately $2\frac{1}{2}$ metres diameter.

Maximum Speed. 300 R.P.M.

Control. Automatic.

Output. 8 tons per machine, per hour.

Charges Per Hour. 3.

2. On refined bi-carbonate.

Maker. Escher Wyss.

Type. Similar in type to (1) above.

Technical Data (Contd.)

Number Installed. 1.

Basket. Approximately 1.7 metres diameter.

Maximum Speed. 400 R.P.M.

Control. Automatic.

Output. 3 tons per hour.