24-030.

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CABLES & TELEGRAMS: MINOILSEP-AVE, LONDON.

CODE: BENTLEY'S.

TELEPHONE: CLERKENWELL 1016.

MINERAL OILS EXTRACTION LIMITED.

(CROZIER RETORT & CONDENSER PATENTS)

DIRECTORS:
SIR T. R. WYNNE, K.C.S.I., K.C.I.E.
DR J. W. GREGORY, F.R.S.
R. H. CROZIER, M.A.I.M.M.E.
H. R. EDWARDS.

FINSBURY COURT,
FINSBURY PAVEMENT,
LONDON, E.C.2.

TESTING PLANT,
BURMA SECTION,
BRITISH EMPIRE
EXHIBITION,
WEMBLEY.

REF.

REPORT ON THE TREATMENT OF TASMANIAN SHALE.

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SUMMARY OF REPORT.

Oil content of shale as determined in Laboratory Retort.

Bulk Sample	No. 1.	46.3	gallons per ton.
" "	No. 2.	46.0	" " "
" "	No. 3.	44.7	" " "
Average.		45.67	" " "

Engler Distillation of Laboratory Crude Oil.

I.B.P. 70° C.		
Under 130° C.	2.0%	No. 1. Petrol.
" 175° C.	4.5%	No. 2. "
" 250° C.	8.6%	No. 1. Kerosene.
" 300° C.	11.9%	No. 2. "
Over 300° C.	73.0%	Fuel Oil or Lubricating Oil Base and Pitch.

Results from treatment of 6tons. 13 cwt. of the shale in the 10 ton unit of the "Crozier" Retort.

Recovered Oil.	280 gallons
Gallons per Ton.	42.1

Recovery based on Laboratory determination of Oil content of shale. 92.1%

Crude Oil bulked into four fractions as follows.

Fraction A.	6.0 %	Crude	Sp.Gr.	.855
" B	3.32%	"	" "	.880
" C	7.33%	"	" "	.920
" D	83.34%	"	" "	.965
	100.00			

A, B and C Fractions consisting of 16.66% of the Crude Oil Contain all the No. 1. Motor Spirit and 90.3% of No. 2. Motor Spirit.

It is anticipated that in commercial operation completely fractionated products will be recovered in a condition easy to refine into marketable grades and in consequence an economy in refining operating costs and capital expenditure amounting to some 80% may be affected as compared with other methods of shale treatment.

Estimated Treatment Costs. - on 500 ton a day basis.

	s.	d.
Mining..............	8.	0
Breaking............		3
Retorting...........	2.	0
Condensing..........		3
C/F	10.	6

	s.	d.
B/F	10.	6.
Refining..................		8.
Marketing.................	1.	6.
	12.	8.

<u>Capital Charges against operating.</u>

Amortization...............		10.4
Interest...................		8.3
Insurance..................		4.0
	14.	6.7

Estimated cost of complete Plant to treat 500 tons per day

£65,000.

Estimated Gross Value of Products from 1 ton of Shale.

	Yield Gals.	Price.	Value Per ton Shale. S.	d.
Motor Spirit.	5.73	2/- per gal.	11.	6.
Kerosene.	8.64	1/4 " "	11.	6.
Fuel Oil.	25.20	£6. " ton	12.	9.
Pitch.	5.46	£3. " "	1.	5.
			37.	2

<u>Estimated Profit.</u>

Gross Value Products.	£1. 17. 2.
Less Operating Costs.	14. 7.
Estimated Profit.	£1 2. 7.

The treatment of the shale and the refining of the oils produced offer no difficulties in our apparatus and there is every reason to expect that the Industry may now be established on a highly payable basis.

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DESCRIPTION OF PROCESS.

In order that the details of the Report may be clearly understood it would appear desirable at this point, to give a description of the apparatus and process in which the tests have been carried out.

The Retort.

In the accompanying blue print of the plant it will be seen that the Retort proper is constructed in the form of a flat sided cylinder with semi-circular ends. This retort is surrounded by brickwork, constructed so as to form an encircling external flue which is divided into compartments by means of fire tile partitions The upward passage of the gasses from the furnace, through the various compartments of these external flues is controlled by means of dampers which may be operated in such a way as to permit a straight ascent of gasses to the top of the retort on either side, or the heat may be forced to encircle the retort and traverse each compartment of the external flues before reaching the chimney.

The Retort proper is provided with internal flues which traverse the apparatus diagonally. These flues conduct the heat across the retort to the opposing external flues and serve as a means of distributing heat throughout the descending mass of material under treatment. The internal flues are provided with dampers and it will be observed that by this system of dampers and flues the temperature may be effectively controlled in each section of the retort.

In the process of treatment the material, broken to a suitable size (in the present test the shale was broken in pieces of from 3 to 4 inches in size and charged containing approximately 10% of material which would pass through a $\frac{1}{4}$ inch mesh screen) is charged continuously in at the top of the retort. The shale flows freely by gravity through the retort and is discharged by means of a screw at the bottom, the rate of feed being regulated by the discharge.

As the shale becomes heated in its descent through the retort the oil content is yielded progressively with the increase of temperature and a primary rough fractionation of the constituents is thus affected. In order to take advantage of this fractionation and to obviate the possibility of internal condensation and 'cracking' of the vapours the apparatus is provided with a series of vapour traps, arranged at different levels by means of which the vapours are trapped and withdrawn from the retort immediately on formation and at their respective formation temperatures. Each of the series of vapour take-offs leads to a separate condenser. The vapours are drawn by means of a fan through the condensers in which the oils are collected. After the elimination of the oil vapours, the fixed gas passes through the fan and in a commercial plant would be forced through scrubbers for the removal of the lightest spirit and ammonia and then to the retort fire-box to be burned to provide the heat for the process. The fixed gas from the Tasmanian shale should be sufficient to supply the heat necessary for the retorting process, which should be entirely self-supporting as regards to heat once the retort is in proper operation.

The Condensers.

The condensers consist essentially of oblong steel boxes to the cover plates of which are attached the series of inverted U tubes or pipes in which condensation of the vapours takes place by means of atmospheric cooling. The condenser boxes are divided into a series of compartments which separately collect the condensed products from each of the ascending and descending pipe columns. They are also provided with a series of baffles in the form of traps, which while forcing the vapours to travel through the cooling pipes, do not prevent the free flow of the condensate from the cool end, toward the hot end of the apparatus.

At the Wembley plant each of the four condensers contains eight compartments. These compartments being numbered from the cool end of the condensers. No. 1. therefore receives the lightest condensate while No. 8. (boiling box) receives the hot incoming vapours direct from the retort.

The boiling boxes are constructed so as to act in the manner of stills and the incoming vapours are trapped and boiled through the primary condensate, which is maintained at a definite level in these boxes. In this manner the immediate condensation of only the highest boiling point constituents of the vapours is effected and the vapours which travel forward and condense in the body of the condenser are of the character of distillates. In the arrangement of the condensers a difference in level is provided which permits the excess condensate from the boiling box of No. 1. condenser to flow continuously to the boiling box of No. 2. condenser and so on down the series, giving up its lighter constituents in succession, according to the increasing boiling temperature to which it is submitted. The arrangement also permits the gravitational flow of the light condensates in the No. 1. boxes back to the boiling box of the preceeding condensers, and the condensates in the intermediate compartments overflow and cascade back to the boiling boxes of the individual condensers. It will be seen from this counter current cascade arrangement, which is entirely automatic in operation, that all the lightest fraction may thus be concentrated in the No. 1. box of No. 1. condenser and that all the highest boiling point oils are concentrated in the boiling box (No.8.) of No. 4. condenser while different grades of Kerosenes, fuel oil or lubricating base are taken off from the intermediate compartments.

Fractionation.

The extent of the fractionation effected by this arrangement may be gauged from the Specific Gravities of the oils recovered from the various boxes of the condenser when treating the Tasmanian shale.

Specific Gravities of Tasmanian Shale.
Crude Oil from Condensers.

Box	Condenser 1	Condenser 2	Condenser 3	Condenser. 4
1	.882	.840	.847	.865
2	.900	.856	.853	.888
4	.912	.866	.880	.897
6	.940	.915	.914	.925
8	.950	.957	.970	.985

The unsymetrical character of the figures from No. 1 condenser is due to the boiling box of this condenser not having been in effective operation throughout the whole of the run, thus permitting the primary vapours to go forward into the condenser without trapping, giving the relatively higher gravity figures shown. It is intended in larger scale continuous operation, to maintain the respective temperatures of the boiling boxes if necessary by means of auxillary external heating. The fractionation shown in the above figures has been effected entirely by the excess and latent heat in the vapours coming from the retort. The figures leave no doubt, however, that under continuous commercial operation a complete fractionation of the oil, according to the boiling point of its various constituents may be effected.

Dissection of Engler Distillations of Recovered Fractions.

The crude oil recovered throughout the test was bulked into four main fractions having the characteristics shown in the table attached.

The extent to which the fractionation has been carried in the present test may be clearly seen from these figures.

It will be observed that the dissection of these figures shows that 76% of the No. 1. Petrol, boiling under 130° C. was concentrated in the fraction A, which consisted of 6% of the total recovered oil, and that this fraction contained only 4% of the oil boiling over 300° C. On the other hand fraction D contains none of the No. 1. Petrol and only .52 of the No. 2. Petrol. It consists chiefly of high boiling point oils and contains over 95% of the oil boiling over 300° C.

The difference in the percentage of the low-boiling-point fraction in the cal ulated composition of the crude oil as compared with the Engler determination of the crude oil produced under laboratory conditions is due to the comparative ineffiency of the plant condenser as compared with the laboratory condenser. This deficiency would of course, be provided for in a commercial plant

Characteristics of Recovered Fractions.

		Fraction A.	Fraction B.	Fraction C.	Fraction D.
% Crude Oil.		6.0%	3.33%	7.33%	83.34%
Specific Gravity.		.855	.880	.92	.962
Engler Distillation.					
Under 130° C.)	Petrol	17.0%	5.0%	2.0%	nil.
" 175° C.)		44.0%	34.0%	5.0%	.5%
" 250° C.)	Kerosene.	25.0%	35.0%	42.0%	11.0%
" 300° C.)		10.0%	14.0%	21.0%	19.0%
Over 300° C.	Fuel Oil and Pitch.	4.0%	12.0%	30.0%	69.5%
I.B.P. 60° C.					

Dissection of Engler Distillation of Recovered Fraction.

			~~Under 130° C.~~ Under 130°C			Under 175° C.			Under 250° C.			Under 300° C.			Over 300° C.		
Fraction.	Sp.Gr.	% Total Oil.	Engler %	% Total Oil.	% Total Fraction.	Engler %	% Total Oil.	% Total Fraction.	Engler %	% Total Oil.	Distribution.	Engler %	% Total Oil.	% Total Fraction.	Engler %	% Total Oil.	% Total Fraction.
A.	.855	6.0	17.0	1.02	76.1	44.0	2.64	57.6	25	1.50	10.1	10	.60	3.2	4	.24	.39
B.	.880	3.33	5.0	.17	12.7	34.3	1.13	24.6	35	1.17	7.8	14	.47	2.5	12	.40	.66
C.	.920	7.33	2.0	.15	11.2	5.0	.37	8.1	42.0	3.08	20.7	21	1.54	8.4	30	2.2	3.62
D.	.962	83.34	-			.5	.44	9.7	11.0	9.17	61.4	19	15.83	85.9	69.5	57.92	95.33
Calculated Engler.				1.34			4.58			14.92			18.44			60.76	

Fractions A, B and C = 16.66% of Total crude oil.
Contain all oil Boiling under 130° C.
" 92.5 of oil boiling under 175° C.

Fractions A and B = 9.33% of Total crude oil.

contain 88.8% of oil Boiling under 130° C.
" 83.7% " " " " 175° C.

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Commercial Aspect of our Refining Proposals.

In commercial practice with a fully equipped condenser section the temperatures of the boiling boxes would be maintained as follows

No. 1. Condenser	150° C.
No. 2. "	225° C.
No. 3. "	275° C.
No. 4. "	350° C.

Practically all of the heat necessary to maintain these temperatures would be conveyed by means of the vapours direct from the retort. The plant design, however, would provide for additional heat to be applied to the "boiling boxes" should such be found necessary for the process. In this manner with the condenser equipped as suggested the following completely fractionated oils would be recovered direct from the shale in one operation.

Fraction.	B.P.	Remarks.
No. 1. Petrol	Under 130° C.	Semi-Refined.
No. 2. "	130° - 175°	" "
No. 1. Kerosene	175° - 250°	" "
No. 2 "	250° - 300°	" "
Fuel Oil or Lubricating Base.	300° - 350°	Sufficiently refined or Fuel Oil purposes.
Pitch	Over 350° C.	Marketable as such.

The amount of subsequent refining which would be necessary to make marketable products of those fractions which are marked semi-refined has not yet been determined but laboratory work on the oils produced in this test indicate that they would require only filtration through bauxite, silica gel or other decolourizing medium to put them in a condition suitable for the market.

The fuel oil and the pitch fraction may be marketed as such immediately on production.

It will be seen therefore that the adoption of this process of retorting and immediate fractionation, would insure a reduction in the capitalization costs of refining plant by some 80% as compared with the ordinary methods of treatment which depend upon the production of one bulk crude oil. In addition the operating costs of refining are practically eliminated.

The statements made in the preceeding paragraph, are based on the results obtained at our Wembley Plant and from these results we consider that the refining plant would be limited to apparatus for the handling of the scrubber oil for the recovery of the light spirit, and a filtering section for the treatment of the products recovered from the main condensers.

Provided, of course, that it is not decided to undertake the manufacture of lubricating oils in which case the necessary refining plant for this purpose would have to be installed.

Our proposals in connection with the refining of the oil will be seen more clearly from an examination of the oil samples which accompany this report.

Samples A, B, C and D represent the four main fractions into which the crude oil has been divided.

Samples A 1. and A 2. have been obtained by one laboratory distillation of sample A. It is in this stage of refinement that we would expect to recover these oils under continuous operating conditions.

Samples A 3. and A 4. have resulted from the redistillation of A 1. and A 2. Samples A 5. and A 6. are fractions A 1. and A 2. which have been given 1% acid treatment and redistilled. These fractions represent refined spirits of good quality.

It will be seen that the amount of refining necessary to produce refined spirit is very little and the results indicate that this may be considerably reduced on practice.

The possibility of producing clean oils direct from fractions A 1 and A 2. by means of bauxite or silica gel filtration has not yet been fully investigated. Preliminary results, however, using a bauxite sample of indifferent quality indicate that the colour of these oils may be completely removed by such treatment.

Mining Costs.

For the prupose of this report I have estimated 8/- per ton as the cost of mining 500 tons per day of the Tasmanian shale and and delivery to the breaker station hoppers.

For the purpose of breaking, toothed rools should be installed of a capacity sufficient to handle in one shift all the shale required for the 24 hours operation.

The shale would broken to a maximum size of 6 inches and stored, to be drawn as required, from the breaker station bins.

The cost of breaking as indicated would not exceed 3d per ton.

	Per ton shale.
Mining	8. 0.
Breaking............................	3.

Retorting Costs.

Under this heading will be included all working charges incidental to handling 500 tons per day of the broken shale from the breaker station bins to the retort and for the disposal of the spent residue to the dumps.

These charges are naturally dependant to a large extent upon the general lay-out and design of the plant. The scheme in mind, however, presupposes that it will not be possible to secure a gravity flow of the material through the plant, but that some means of elevating to the retort feed hoppers will have to be provided for and also that it will be necessary to stack the residue above the prevailing level of the country. These charges for handling, which would vary according to the method adopted, while not strictly retorting charges, are best included under this heading.

	Per ton shale.
Cost of conveying to and charging Retorts.	0 - 2
Retorting.	
Labour. 4 men per 8 hr. shift at 15/-	
3 Foremen at 20/-	0 - 6
Power. 10 h.p at 2d per h.p.h.	1
Steam to Retort.	1
R & M Charges. ½ Overhead	
½ Laboratory. ¼ Lighting Charges etc.	1.- 0
Disposal of Residue.	2
	2. 0.

Condensing Charges.

Two men per shift with the supervision of the foreman would be sufficient for the control of the condensing section of the plant. In addition to the labour charges there would also be the cost of operating the fan which would be chargeable under this heading.

	Per ton shale.
Labour. 6 men at 15/-	2.
Power. 3 h.p. at 2d per 1 h.p.h.	.25
Supervision. Laboratory R & M.	.75
	3

Refining.

Charges under this heading would be contingent on the marketing conditions decided upon. It is anticipated that under steady running conditions it will be possible to produce some 30% of the oil in Petrol and Burning Oil fractions which would require only little subsequent refining treatment to be made of marketable quality.

The cost of such treatment which may in an extreme case include a light acid treatment, one distillation and bauxite filtration, should not exceed ½d. per gallon. Thus on a recovery

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of 12 gallons per ton of these fractions the cost would be 6d per ton shale.

Refining Costs. Light Fractions.

In addition to the above refining costs would have to be included the cost of recovering and refining the light spirit from the fixed gas.

This spirit is recovered by scrubbing the gas in a tower by means of heavy oils which act as a medium for absorbing the light hydrocarbons. The spirit being subsequently recovered from the heavy oil by steaming. The process is continuous and simple in operation.

Accurate data for estimating the cost of recovering the spirit from the gas of the Tasmanian shale is not available, since absorption apparatus for this purpose has not been installed at our Wembley Plant.

We should expect, however, that the oil to be recovered in this manner would amount to some 3 gallons per ton of shale treated. The cost of operating this section of the plant and of refining the recovered spirit would not exceed 2d per ton shale.

We therefore have.

	Per ton shale.
Refining of Petrol and Kerosene Fractions. and Recovery of Scrubber Spirit.	8d.

Summary of Working Costs.

	Per ton shale.	
Mining and Breaking.	8.	3.
Retorting.	2.	0.
Condensing.		3.
Refining.		8.
Total Working Costs.	11.	2.

Capitalization.

The following figures are given as a rough estimation of the capital outlay involved in commencing operations on a 500 ton a day basis in Tasmania. These figures are based on Rangoon costs of manufacture and their application to Tasmanian conditions, in the absence of detailed drawings and specifications may be entirely misleading. We therefore do not vouch for their accuracy and the estimate is given here merely as a basis on which

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to estimate the capitalization charges, interest, amortization and insurance, chargeable against operating costs.

Mining Plant.	£10,000
Power & Boiler Plant.	10,000
Breaker Station and Transport.	1,500
Retort Plant.	20,000
Condenser Plant.	5,000
Scrubbers.	1,000
Refining Plant.	5,000
Cooperage Canning Plant.	3,000
Storage Tanks.	1,500
Laboratory and Unforeseen.	3,000
Erection.	5,000
	£65,000

Amortization.

Assuming an average amortization charge of 10% on the whole of the above capital outlay, then we have a sum of £6,500 per annum chargeable against operating costs. On an average daily throughput of 500 tons, assuming 300 days as a working year the annual throughput of the plant would be 150,000 tons. The amortization charge therefore works out at -

	Per ton shale.
Amortization.	10.4 d.

Interest.

Allowing interest at 8% on an invested capital of £65,000 we have a sum of £5,200 per annum chargeable against operating costs or:-

	Per ton shale.
Interest charge.	8.3

Insurance.

Assuming a valuation of £5000 for insurance purposes as the above capitalization and a premium of 5% on the plant we arrive at a sum of £1250 chargeable against operating cost or:-

	Per ton shale.
Insurance.	4d

Summary of Capital Charges Against Operating.

	Per ton shale.
Amortization.	10.4 d
Interest.	8.3
Insurance.	4.0
Total	1. 10.7
Working Costs.	11. 2
Total Operating Costs.	13. 0.7
Add Marketing and Selling Charges.	1. 6
	14. 6.7

Estimation of Profit.

A recent issue of the Melbourne "Age" indicates the following prices as prevailing in Australia.

Motor Spirit.	2. 6. per gallon.
Kerosene.	1. 8. " "

For the purpose of this report the following prices are taken as abasis of calculation.

Motor Spirit.	2. 0. per gallon.
Kerosene.	1. 4 " "
Fuel Oil.	£5. 0. 0. " ton.
Pitch.	£3. 0. 0. " "

Estimating on the recovery of 42 gallons of oil direct and 3 gallons of scrubber spirit and basing our calculations on the Engler determination of the crude oil composition we have.

Scrubber Petrol.		3 gals	at 2/-	6. 0.
No. 1 and 2 Petrol.	6.5 =	2.73 gals.	at 2/-	5. 6.
Kerosene.	20.5%=	8.61 "	" 1/4	11. 6.
Fuel Oil.	60.0%=	25.2 "	" £5.	12. 9.
Pitch.	13.0%=	5.46 "	" £3.	1. 5.
				37. 2.

Gross Value of Recovered Products from 1 ton of Shale.

	1. 17. 2.
Total Operating Costs.	14. 7.
Estimated Profit.	1. 2. 7.

Conclusion.

The shale as broken by hand at Wembley was somewhat irregular in size. On this account some of the larger pieces (exceeding 4" in size) contained a core of semi-treated material. This, together with the slight inefficiency of the condenser has had the effect of reducing the recovery.

With mechanically broken shale and with the conditions of treatment set to treat pieces of maximum size, and with a completely equipped condensing plant, these sources of loss would be eliminated and we would expect to recover, under working conditions, some 90% of the oil content as indicated by laboratory determination.

The shale is particularly suitable for treatment in our retort and the recovered oil is of a character which lends itself readily to our system of fractionation. We have, therefore, no doubt that our plant can be adapted to handle any daily tonnage which may be

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required.

The method of refining suggested in this report eliminates the factor of refining losses.

The fuel oil fraction and pitch should be marketable as such direct from the condensers. Laboratory work indicates that the Kerosene and Petrol fractions may be refined without acid treatment. Should acid treatment prove necessary then the light acid tars produced could, after treatment for the recovery of the spent acid be incorporated in and sold with the fuel oil fraction.

It is anticipated, however, that filtration treatment will be sufficient for the refining of the light fractions, in which case the extracted impure oils recovered in regenerating the filtering material, should find a ready market as fuel oil.

The margin of profit shown in the report leaves no doubt that the industry should be highly profitable on any thing like the prevailing prices for Motor Spirit, Kerosene, Fuel Oil and Pitch.

Mineral Oils Extraction Limited.

R. H. Crozier

General Manager.

Appendix 1.

There have been various rumours regarding the presence of gold in the Tasmanian shale and it occurred to us that if gold in any appreciable quantity existed in the shale it would probably be associated with the pebbles which are scattered throughout the shale and which show considerable signs of secondary mineralization. Any subsequent effort to make use of the shale residue for the manufacture of cement or other purposes would necessitate the preliminary removal of the pebbles and therefore a possible concentration of gold values.

In order to investigate this point a sample of the pebbles broken from the shale, was sent to Messrs. Sulman & Picard for assay. Their report which is attached, shows only a trace of gold and 2 dwt. 15 grs. silver per ton. The proposition therefore has no commercial value and the matter is mentioned merely as of academic interest.

Appendix 2.

It has been discovered that the shale residue possesses certain properties of a physical -chemical character which give it a value quite apart from any other purpose which has been suggested for its use.

This matter which we regard as of considerable importance has not yet been fully investigated. Work is still in progress however, and we will communicate with you further in this connection when more conclusive data has been obtained.

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SULMAN & PICARD,

Metallurgical Chemists and Assayers.

H. LIVINGSTONE SULMAN, F.I.C., M.I.M.M.
HUGH F. K. PICARD, ASSOC. R.S.M., M.I.M.M.

LABORATORY.

44, LONDON WALL.

LONDON E.C. Nov. 3rd 1924

Certificate of Assay.

Sample of Mineral

Received from R. N. Crozier Esq *Date* 1. 11. 24

Marked "Pebbles from Tasmanian Shale"

~~*Sealed*~~ *Reference No.* G 239

SAMPLE	GOLD			SILVER			—	—	—	—
	Oz.	dwt.	grs.	Oz.	dwt.	grs.	%	%	%	%
as above	trace			0	2	15	per long ton.			

~~*REMARKS*~~

Sulman & Picard

(75)