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ROBERTSON RESEARCH INTERNATIONAL LIMITED

REPORT NO: 5484P/D

RESULTS OF T.O.C. AND "ROCK-EVAL" PYROLYSIS  
ANALYSES OF SIDEWALL CORE SAMPLES  
FROM 30/11-4 WELL, NORWEGIAN NORTH SEA

By

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PROJECT NO: RRPS/845/D/25159

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November 1984.

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SUMMARY

This report contains the results of total organic carbon and "Rock-Eval" pyrolysis analyses of 67 sidewall core samples from the 30/11-4 well. Brief comments are made on the organic richness and maturity of the samples.

## 1. INTRODUCTION

This report contains the results of a series of pyrolysis analyses carried out on 67 sidewall core samples between 3351.5m and 5062m in the 30/11-4 well. Samples were received for analysis on 28th August 1984, with Norske Shell Service Order No. 5 Nr. 5182. Initial results were delivered personally to Mr. Per Møller-Pedersen on October 25th 1984.

The personnel involved during the course of the project were:

Mr. Per Møller-Pedersen of A/S Norske Shell, and Messrs. P. C. Barnard and S. Thompson of Robertson Research International Limited.

## 2. RESULTS

### 2.1 Methods

Small fragments of sidewall cores consisting of mainly dark grey siltstones were received for analysis. These samples were washed and dried at less than 40°C to remove drilling mud contaminants. The lithology of the samples was then briefly described prior to crushing in a disc mill to pass through a 60 mesh sieve. The resulting powders were then analysed for total organic carbon content and pyrolysis yields. None of the samples needed solvent extraction prior to analysis, and the analytical results did not show sufficient free hydrocarbons to interfere with the data.

### 2.2 Presentation of results

The results obtained from the source rock analyses are presented in Tables 1A to 1C. Copies of the pyrograms for the samples are shown in Appendix 3.

### 2.3 Comments on results

The sidewall core series analysed penetrates an often organically rich source rock sequence which, on the basis of the Tmax data, ranges from middle mature (3351.5m to 3384m) to late mature (3384m to 4375m) and post mature (4375m to 5062m).

The samples above 3432.5m show fair to very good oil generating potential. Between 3955m and 4225m the richer intervals have probably generated a fair quantity of oil, and have the potential to generate a minor to fair quantity of condensate. The organically rich samples between 4746m and 4956.5m may have generated oil, condensate and gas during their middle, late and post-mature stages. At present, the horizons at 4933.5m and 4956.5m have the capacity to generate fair quantities of dry gas.

Significant quantities of migrant oil (more than 1000 ppm) are present in the samples from between 4143.5m and 4189m.

GENERAL DATA			CHEMICAL ANALYSIS DATA												
SAMPLE DEPTH (METRES)	SAMPLE TYPE	ANALYSED LITHOLOGY	ORGANIC CARBON % OF ROCK	PYROLYSIS					SOLVENT EXTRACTION						
				TEMP - ERATURE °C	HYDROGEN INDEX	OXYGEN INDEX	PRODUCTION INDEX	POTENTIAL YIELD (ppm)	TOTAL EXTRACT (ppm)	HYDRO-CARBONS (ppm)	EXTRACT % OF ORGANIC CARBON	HYDROCARBONS		ALKANES % OF HYDRO-CARBONS	
													mg/g OF ORGANIC CARBON	% OF EXTRACT	
3351.5	Swc	MDST, ol-blk, mic+ tr SLTST, blk, carb	6.47	439	518	20	.07	33550							
3355.5	"	MDST, a/a	4.26	437	339	21	.10	14460							
3360	"	SH, ol-blk	5.18	438	386	17	.09	20020							
3364.5	"	SLTST, ol-blk	5.07	442	505	21	.08	25650							
3369	"	SLTST, dk gy, carb	4.20	444	484	16	.07	20340							
3372	"	SLTST, blk+ 20% MDST, med-dk gy	3.78	442	396	19	.09	14990							
3375	"	SLTST, brn-blk	3.18	438	316	22	.09	10080							
3379	"	MDST, dk gy, mic	3.44	437	347	17	.09	11950							
3384	"	MDST, dk gy, mic+ 40% SLTST, gy-blk	2.70	440	376	13	.07	10160							
3392	"	MDST, dk gy	2.32	447	265	17	.09	6170							
3396	"	SLTST, brn-blk, mic	2.36	444	213	11	.08	5040							
3403	"	MDST, dk gy	2.66	446	253	16	.08	6750							
3409	"	MDST, med-dk gy	1.57	437	79	54	.08	1250							
3428	"	SLTST, med-dk gy, mic	1.46	442	267	23	.08	3910							
3432.5	"	SLTST, dk gy, mic	2.06	448	223	19	.11	4610							
3477	"	SLTST, pal yel-brn, sndy	.48	443	97	93	.23	470							
3928.5	"	MDST, med-lt gy	.44	448	52	248	.30	230							
3942	"	SLTST, dk gy	1.03	452	89	15	.15	920							
3955	"	MDST, med gy+ tr SLTST, dk gy	3.23	451	47	13	.24	1550							
3989	"	SLTST, med-dk gy, mic	.68	449	75	156	.23	510							
3995	"	SH, dk gy, mic	2.25	451	68	32	.23	1530							
4009	"	SLTST, med-dk gy, mic	1.06	455	92	35	.22	980							
4018	"	SH, med-dk gy	1.48	451	64	17	.25	950							
4043.5	"	SLTST, med-dk gy, calc	1.11	453	54	21	.27	610							
4054.5	"	SH, med-dk gy, calc	1.35	451	56	29	.31	760							
4067	"	SLTST, med-dk gy, calc	1.15	450	53	23	.29	610							
4104.5	"	SLTST, med gy, sndy+ tr mic	.79	450	64	38	.29	510							
4118	"	SLTST, brn-blk, calc	1.02	458	74	85	.30	760							
4131	"	SLTST, ol-blk, calc+ tr SST, pal yel-brn+ tr mic	.91	454	78	41	.30	710							
4143.5	"	SLTST, dk gy, mic	2.15	455	113	56	.33	2440							
4162	"	MDST, med-dk gy, mic	1.62	427	98	45	.05	1590							
4168	"	SLTST, gy-blk, carb	1.81	449	105	17	.41	1910							
4189	"	SH, dk gy+ 10% SLTST, gy-blk	1.85	455	85	17	.41	1590							
4195	"	SLTST, pal yel-brn, mic+ 40% SLTST, gy-blk, calc+ tr mic	.62	449	50	80	.45	310							

Heater

Heater

Chemical Analysis Data

TABLE 1A

GENERAL DATA			CHEMICAL ANALYSIS DATA												
SAMPLE DEPTH (METRES)	SAMPLE TYPE	ANALYSED LITHOLOGY	ORGANIC CARBON % OF ROCK	PYROLYSIS				SOLVENT EXTRACTION							
				TEMP - ERATURE °C	HYDROGEN INDEX	OXYGEN INDEX	PRODUCTION INDEX	POTENTIAL YIELD (ppm)	TOTAL EXTRACT (ppm)	HYDRO-CARBONS (ppm)	EXTRACT % OF ORGANIC CARBON	HYDROCARBONS		ALKANES % OF HYDRO-CARBONS	
													MS/O OF ORGANIC CARBON	% OF EXTRACT	
4225	Swc	SLTST, gy-blk, mic+ 10% SLTST, pal yel-brn, mic	1.80	451	63	34	.37	1140							
4277	"	SLTST, ol-blk+ tr SLTST, pal yel-brn+ tr mic	1.26	455	72	76	.32	910							
4375	"	SLTST, brn-blk+ 10% SLTST, pal yel-brn, sndy	1.27	455	51	57	.40	660							
4407	"	SLTST, brn-blk+ 10% SLTST, pal yel-brn, sndy	1.29	464	48	44	.37	620							
4444	"	SLTST, gy-blk+ mnr SLTST, a/a	1.23	465	37	43	.41	460							
4458	"	SLTST, med-dk gy+ tr SLTST, a/a	1.24	469	47	69	.35	590							
4504	"	SLTST, med-dk gy, mic	.82	472	32	95	.36	270							
4554	"	SLTST, med-dk gy, mic	.95	475	44	68	.32	420							
4581.5	"	SLTST, med-dk gy, calc+ tr mic	.90	470	30	90	.39	270							
4621	"	SH, dk gy, calc	.95	478	21	78	.41	200							
4632.5	"	SLTST, gy-blk, mic	1.03	481	23	16	.26	240							
4690	"	SLTST, pal yel-brn, sndy+ 10% SLTST, gy-blk, carb+ tr mic	.47	498	17	71	.38	80							
4711	"	SLTST, dk gy, mic+ tr SLTST, pal yel-brn, sndy	1.77	485	24	19	.24	430							
4746	"	SLTST, dk gy, carb	4.44	486	38	34	.17	1700							
4786	"	MDST, dk gy, carb	.50	496	23	62	.32	120							
4793	"	SLTST, med-dk gy, carb	3.63	485	36	9	.17	1330							
4815.5	"	MDST, med-dk gy+ tr mic	1.53	495	23	14	.25	360							
4839.5	"	SST, pal yel-brn, slty+ tr mic	.60	*	25	50	.30	150							
4849.5	"	MDST, med-dk gy, carb+ tr mic	.81	*	33	16	.21	270							
4877	"	SLTST, brn-gy, calc+ tr mic	.52	*	*	110	*	*							
4894.5	"	SLTST, dk gy, carb	1.36	501	32	19	.11	440							
4901	"	SLTST, med-dk gy, mic	1.50	507	36	17	.16	540							
4933.5	"	MDST, gy-blk, carb	26.32	504	39		.06	10520							
4943.5	"	MDST, brn-gy, mic	1.01	501	34	23	.17	350							
4956.5	"	COAL	15.10	502	40	2	.05	6170							
4973	"	SH, blk, carb+ tr SST, pal yel-brn, mic+ tr pyr+ tr COAL	1.77	496	25	25	.11	460							
4997.5	"	MDST, med-dk gy, carb+ tr mic	.58	499	31	68	.10	180							
5003.5	"	SST, brn-gy, slty	.66	*	21	71	.30	140							
5011	"	SLTST, brn-gy, carb+ tr mic	1.08	427	46	794	.15	500							
5033.5	"	SLTST, dk gy, carb+ tr mic	.82	*	1	32	.85	10							
5036.5	"	MDST, brn-blk, carb	.66	*	13	265	.38	90							
5052	"	SLTST, brn-blk, carb+ tr mic	.49	*	53	687	.16	260							

Chemical Analysis Data

TABLE 1B

GENERAL DATA			CHEMICAL ANALYSIS DATA												
SAMPLE DEPTH (METRES)	SAMPLE TYPE	ANALYSED LITHOLOGY	ORGANIC CARBON % OF ROCK	PYROLYSIS				SOLVENT EXTRACTION							
				TEMPERATURE °C	HYDROGEN INDEX	OXYGEN INDEX	PRODUCTION INDEX	POTENTIAL YIELD (ppm)	TOTAL EXTRACT (ppm)	HYDRO-CARBONS (ppm)	EXTRACT % OF ORGANIC CARBON	HYDROCARBONS mg/g OF ORGANIC CARBON	% OF EXTRACT	ALKANES % OF HYDRO-CARBONS	
5062	Swc	SLTST, dk gy, mic	.91	*	7	46	.39	70							

Chemical Analysis Data

TABLE 1C



APPENDIX 1  
ABBREVIATIONS USED IN ANALYTICAL DATA SHEETS

a/a	-	as above	MDST	-	mudstone
Ac	-	acritarchs	med	-	medium
ADD	-	mud additive	MET	-	metamorphic rocks
Al	-	algae	mic	-	mica/micaceous
Am	-	amorphous	micr	-	micritic
ang	-	angular	min	-	mineral
ANH	-	anhydrite	mnr	-	minor
aren	-	arenaceous	mod	-	moderate
arg	-	argillaceous	mtl	-	mottled
BAS	-	basalt	n-	-	normal
bd	-	bedded/bedding	NA	-	not available
B(IT)	-	bitumen/bituminous	nod	-	nodule/nodular
bl	-	blue	NS	-	no sample
bld	-	bleached	occ	-	occasional
blk	-	black	ol	-	olive
bri	-	brilliant	ool	-	oolitic
bra	-	brown	orng	-	orange
calc	-	calcareous	OS	-	oil stain
CALT	-	calcite	P	-	picked lithology
carb	-	carbonaceous	pal	-	pale
CGL	-	conglomerate	Ph	-	phytane
CHK	-	chalk	pnk	-	pink
CHT	-	chert	por	-	porous/porosity
CLYST	-	claystone	PP	-	purple
CMT	-	cement	Pr	-	pristane
Comp	-	composite	pred	-	predominantly
crs	-	coarse	Prt	-	present
CSG	-	casing point/shoe	PYR/pyr	-	pyrite/pyritic
Ctgs	-	ditch cuttings	QTZ(T)	-	quartz(ite)
Cu	-	cuticle	Re	-	resin
C(vd)	-	caved	R(ew)	-	reworked
decarb	-	decarbonated	rnd	-	round(ed)
Di	-	dinocysts	Sap	-	sapropel
dk	-	dark	sbnng	-	subangular
DLT	-	dolêrite	sbrd	-	subrounded
DOL/dol	-	dolomite/dolomitic	SCI	-	spore colour index
dsk	-	dusky	Sf	-	semifusinite
Ex	-	exinite	sft	-	soft
Exs	-	exsudatinite	SH	-	shale
extr	-	extracted	shly	-	shaly
f	-	fine	sil	-	siliceous
fel	-	feldspathic	sks	-	slickenside surface
fer	-	ferruginous	SLA	-	slate
flu	-	fluorescence	SLT(ST)	-	silt(stone)
fm	-	formation	slty	-	silty
foss	-	fossils/fossiliferous	SND	-	sand
fr	-	friable	sndy	-	sandy
frac	-	fracture	Sp	-	spores
frags	-	fragments	SST	-	sandstone
Fu	-	fusinite	st	-	stained
GLC/glc	-	glauconite/glauconitic	stks	-	streaks
gn	-	green	suc	-	sucrosic
grd	-	graded/grading to	surf	-	surface
grns	-	grains	SWC	-	side wall core
gy	-	grey	TD	-	total depth
GYP	-	gypsum	TOC	-	total organic carbon
HAL	-	halite	tr	-	trace(s)
hd	-	hard	trns	-	transparent
hor	-	horizontal	v	-	very
H(RV)	-	high reflecting vitrinite	vgt	-	variegated
i-	-	iso-	Vit	-	vitrinite
i/b	-	inter-bedded	vn	-	vein
IGN	-	igneous rocks	VOLC	-	volcanic rocks
inc	-	including	VR	-	vitrinite reflectivity
Inert	-	inertinite	wht	-	white
lam	-	laminae/laminated	xln	-	crystalline
LCM	-	lost circulation material	yel	-	yellow
LIG/Lig	-	lignite/lignitic		-	
lns	-	lens(es)		-	no analysis carried out
L(RV)	-	low reflecting vitrinite	*	-	analysed but no data obtained
LST	-	limestone	gy-gn	-	greyish green
lt	-	light	gy/gn	-	grey-green (gradation)
mass	-	massive	gn-gy	-	greenish grey

Note: (Maturity data tables only). Number in brackets refers to number of reflectivity values averaged to give quoted result. Preferred values for indigenous phytoclasts are listed first.

## APPENDIX 2

### ANALYTICAL PROCEDURES AND TECHNIQUES

This appendix summarises the main steps in the analyses carried out in the Robertson Research International Ltd. petroleum geochemistry laboratories. Analytical pathways are shown on the flow chart (Appendix Figure 1) and details of laboratory procedures and techniques are given in the text. These may in certain circumstances be adapted to suit particular samples or conditions. Interpretation guidelines are also defined.

#### 1. Sample Preparation

##### General

Samples are received into the laboratories in the forms of well-site canned ditch cuttings, bagged ditch cuttings in various stages of preparation from wet, unwashed to dried, washed; sidewall cores, conventional cores, outcrop samples, crude oil samples and gas samples. Each sample is assigned a number which is entered into a computer system to monitor sample selection and progress. Preparation techniques are directed towards obtaining clean samples, free of drilling mud and mud additives, obvious caving contamination and indeterminate fine material. Washing with cold water is standard but further washing with solvent (dichloromethane, DCM) is carried out if oil-based mud is present, after which samples are dried, described and individual lithologies hand-picked where practicable. Samples are rough crushed to approximately pea-sized fragments for kerogen preparation or finely milled for chemical analysis.

##### Kerogen Preparation

Kerogen concentrates for microscopic examination and elemental analysis are prepared using standard palynological procedures but omitting oxidation or acetolysis. Acid maceration involves the use of hot hydrochloric acid (HCl) to remove carbonates and hot 60% hydrofluoric acid (HF) to remove or break down silicates. Mineral residues are separated from the kerogen by a combination of ultrasonic vibration and zinc bromide flotation. Kerogen samples for spore colour and kerogen typing are mounted on glass slides in glycerin jelly, those for vitrinite reflectivity are dried and mounted in epoxy resin. Kerogen residues are stored in methanol.

#### 2. Maturity Evaluation

The techniques employed for interpreting maturity and thermal history in these laboratories are based mainly on spore colouration and vitrinite reflectivity measurement, supplemented by data obtained from airspace gas and gasoline analysis, pyrolysis T<sub>max</sub>, and hydrocarbon analysis including gas chromatography and gas chromatography-mass spectrometry.

##### Spore Colouration

Sporomorph colour is assessed using a >20 $\mu$  sieved kerogen fraction viewed in transmitted light on a standard palynological microscope. Unusual hues are checked using incident blue/UV light fluorescence. Measurement is made by eye against reference sets of single grain spore mounts and trained operators achieve a high degree of accuracy and reproducibility. The 1 to 10 Spore Colour Index (SCI) scale was designed for linearity with increasing depth and temperature and correlates approximately with the following zones of oil generation: 1.0 to 3.5, immature; 3.5 to 5.0, early mature, generation of low gravity oils (28 to 35 °API); 5.0 to 7.0, middle mature, generation of medium gravity oils (35 to 42 °API); 7.0 to 8.5, late mature, generation of light oils (>42 °API) and condensates; 8.5 to 10, post mature, generation of condensate, wet gas and, ultimately, dry gas. Linearity of scale is of great value in prediction, by extrapolation, of the depth to any part of the oil generation sequence. The value of SCI measurement lies in the objective selection of measured grains, so minimising problems of caving and reworking, and in its more direct correlation against oil generation than vitrinite reflectivity measurement. Limitations in its use concern the difficulty of correlation against other colour scales and the insensitivity of the scale in the late to post mature region. Anomalous colours may result from bleaching or staining during deposition and diagenesis. The correlation of SCI against Thermal Alteration Index (TAI) given on the SCI versus depth plot in the reports was made by direct comparison of Staplin's standard slides with SCI standard slides.

##### Vitrinite Reflectivity

The majority of preparations examined under reflected light in these laboratories are made using >20 $\mu$  sieved kerogen, mounted in resin blocks and polished with carborundum and alumina although total kerogen may be used when sample size is

limited. Picked coals, organic-rich shales or limestones containing solid bitumen are mounted directly in resin blocks and polished in the usual way. Measurement is made on a Leitz Orthoplan microscope fitted with an MPV Compact photometer which feeds values direct to a desk top computer for data processing from each sample. The system is calibrated against glass standards and reflectance values are expressed as arithmetic means of measurements taken in oil immersion ( $R_o$  or  $R_{m\ oil}$ ).  $R_{max}$  and  $R_{min}$  may be measured and quoted in certain circumstances but the difference is insignificant below about  $R_o$  1.0%. Some operator selection of particles during measurement is essential and obvious contaminants or non-vitrinitic material are noted but not necessarily quoted. The value quoted on data tables is that which is interpreted as most appropriate, but other possibilities may also be given. Plotted figures assume a logarithmic increase of reflectance with depth.  $R_o$  0.5% is a widely accepted threshold value for the onset of oil generation, although as the kinetics of oil generation may not be identical to those of vitrinite reflectivity development this must be seen only as a general guide. The floor for oil generation is characterised by a reflectance value of about 1.3%. Wet gas generation peaks at a value of about 1% and ceases at the 2% level. Dry gas generation peaks at a reflectance of about 1.5% and ceases at the 3% to 4% level. Correlation of reflectance values with other maturity parameters may not be universal because of time-temperature factors and is best made on a local basis.

Reflectivity measurement is a widely used and versatile tool which may be readily calibrated against easily obtained standards. It is applicable over a wide range of maturity stages from immature to post mature (0.2% to 5%  $R_o$ ). High surface intercepts on plotted figures and discordances at faults and unconformities can give realistic estimates of the amount of section missing. It is of limited value in Early Palaeozoic sections where land plant material is absent, although a general guide to maturity may be obtained from chitinous organic matter. Even a skilled operator may have difficulty in distinguishing indigenous vitrinite from some forms of inertinite, anomalously reflecting "pseudovitrinite", cavings and reworked fragments.

#### Airspace Gas Analysis

Wet cuttings are collected at the well site and sealed in partly full cans containing bactericide. In the laboratory, the airspace (headspace) gas is extracted using a can piercer fitted with a septum and analysed by gas chromatography. The proportions of methane, ethane, propane, iso- and n-butane are calculated from integrated peak areas by comparison with a standard mixture of these gases. Methane is the dominant gas in immature and post mature sediments, comprising 90-100% of total gas, falling to 30-70% in mature sediments. The onset of maturity for oil generation (SCI 3.5) is characteristically marked by an increase in wet gas ( $C_2-C_4$ ) to between 10 and 20% with further increases in maturity indicated by a decrease in the ratio of iso- to n-butane. Ratios of >1.0 are typical for immature sediments and <0.5 are usual in mature sediments. Departures from composition versus depth trends may be useful in indicating migrant gas at faults, unconformities or reservoir rocks but limit the method as a reliable maturity indicator. Airspace gas analysis is an inexpensive and rapidly executed method of screening samples for further maturity and hydrocarbon content determinations.

#### Gasoline Analysis and Cuttings Gas Analysis

Cuttings samples received wet, preferably in sealed containers, are suitable for gasoline and cuttings gas analysis. A portion of the washed cuttings sample is retained wet, pulverised in a sealed shaker and warmed to expel the  $C_1$  to  $C_7$  hydrocarbon components into the shaker airspace. A sample of this airspace gas is then removed and analysed by gas chromatography either for cuttings gas ( $C_1$  to  $C_4$ ) or gasolines ( $C_4$  to  $C_7$ ). Up to 28 hydrocarbon components are identified in the  $C_4$  to  $C_7$  range and their relative proportions calculated from integrated peak areas with reference to standard mixtures. Immature source rocks yield low total abundances and limited numbers of components whereas mature source rocks usually contain a full complement of identified hydrocarbons with the onset of maturity indicated by a rapid rise in total gasoline abundances with depth. Anomalous amounts of gasolines may mark the presence of oil stain. Gasolines may be used in oil to oil or oil to source rock correlations but the concentration of some of the measured components is not only a function of source but also depends on maturity, migration and alteration in the reservoir. Using the most stable compounds, pairs with similar chemical structure and boiling points are reduced to pair ratios and compared with the same pair ratios in other oils or possible source rocks. Gasoline analysis is a valuable tool in that it measures directly the hydrocarbons being generated from a sediment but its sensitivity in detecting traces of oil places constraints on its use as a maturity indicator.

Rock-Eval Pyrolysis, Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometry (GC-MS) in Maturity Analysis  
These three analytical processes measure parameters which are functions of both maturity and kerogen type. Data from them may give a general guide to maturity but if the kerogen types are known, more specific conclusions may be drawn. From Rock-Eval data, the temperature of maximum rate of pyrolysis,  $T_{max}$ , is the most useful datum; gas chromatograms of alkanes, separated from source rock extracts or oils, yield carbon preference indices (CPI) and isoprenoid ratios; GC-MS quantitative fragmentograms provide abundance ratios for specific compounds which are particularly useful in assessing the level of maturity at which source rock hydrocarbons or oils have been generated. All these supplementary data may be used to confirm results from visual analysis or supplant them if poor or unavailable.

### 3. Source Rock Evaluation

#### Total Organic Carbon Content (TOC)

Organic carbon values are obtained by treating 0.1g of crushed rock sample with hot, concentrated HCl to remove carbonates. The washed residue is filtered on to a glass fibre pad and ignited in a Leco carbon analyser. For screening purposes, samples are analysed singly but where further analyses, such as pyrolysis or solvent extraction are anticipated, a duplicate sample is run. Blanks and standards are run as routine and where values from duplicated samples do not concur within strict accuracy limits, they are rerun. Where samples are heavily stained with oil, either from natural deposits or drilling mud, TOC is repeated on the dried, solvent extracted sample.

TOC measurement is fundamental in assessing source rock quality since when combined with kerogen type and maturity, a full description of the potential to generate oil may be given. It is found in practice that sediments containing less than 0.3% TOC are unlikely to have any source potential, those containing between 0.3% and 1% may be marginal sources but the better quality sources contain in excess of 1% TOC. Screening by TOC is therefore an inexpensive and rapid method of selection of samples for further analysis in source potential evaluation.

#### Rock-Eval Pyrolysis

Pyrolysis data are obtained using the IFP-Fina Rock-Eval apparatus. 100 mg of crushed, whole rock either from bulk sample or picked lithology is weighed accurately into a crucible and introduced into a furnace at 250°C. Free hydrocarbons (roughly equivalent to solvent extractable hydrocarbons) are volatilised and quantified by flame ionisation detector (FID) to give Peak 1 ( $S_1$ , ppm). The furnace temperature is increased to 550°C at 25°C/minute and within this range, kerogens crack to give hydrocarbons, measured by FID to give Peak 2 ( $S_2$ , ppm) and carbon dioxide, measured by thermal conductivity detector (TCD) to give Peak 3 ( $S_3$ , ppm). The temperature at the maximum rate of evolution of cracked volatiles ( $T_{max}$ ) is measured automatically but can also be monitored visually. The instrument is calibrated daily using standards both at the beginning of the work period and at regular intervals thereafter and crucible blanks are run as routine. The tabulated data in reports comprise the following parameters:

- $T_{max}$  °C - temperature of maximum rate of Peak 2 hydrocarbon evolution.
- Hydrogen Index (HI) -  $S_2/TOC$  (mg/g) or ratio of released hydrocarbon to organic carbon content. This is a measure of the hydrocarbon generating potential remaining in the kerogen as opposed to that of the whole rock.
- Oxygen Index (OI) -  $S_3/TOC$  (mg/g) or ratio of released carbon dioxide to organic carbon content.
- Production Index (PI) -  $S_1/S_1+S_2$ , or ratio of the amount of hydrocarbons released in the first stage of heating to the total amount of hydrocarbons released and cracked during pyrolysis.
- Potential Yield (PY) -  $S_2$  (ppm) or total of hydrocarbons released during cracking of kerogen compared to original weight of rock.

$T_{max}$ , hydrogen index and oxygen index are each functions of both maturity and kerogen type. Using published and empirical data, it has been possible to assemble a model to show the relationships of these factors to maturity as measured by spore colouration and vitrinite reflectivity for a selection of pure kerogen types. The kerogen types used are algal sapropel (type I), waxy sapropel (type II), vitrinite (type IIIA) and inertinite (type IIIB) and a computer program has been devised by which the amounts of these components may be calculated from the HI, OI,  $T_{max}$  and maturity

data for any sample. These are the values expressed in the "kerogen composition by calculation" columns tabulated in the reports.

The hydrogen index is a measure of the hydrocarbon generating potential of the kerogen and is analogous to the atomic H/C ratio. Immature, organically rich source rocks and oil shales give values above 500, mature oil source rocks give values between 200 and 550. For a given kerogen type, these values progressively diminish with increasing maturity.

The temperature of maximum rate of pyrolysis depends partly on the kerogen type but the transition from immature to mature organic matter is marked by temperatures between 415° and 435°C. The maturity transition from oil and wet gas generation to dry gas generation is marked by temperatures between 455° and 460°C. In practice, greater variation than these ideal temperature ranges may be seen, but they are nevertheless useful as general guides to the level of maturity attained by the sediment.

The production index increases with maturity from values near zero for immature organic matter to maximum values of 0.15 during the late stages of oil generation. Anomalously high values indicate the presence of oil or contaminants. The potential yield is an indication of the predicted yield of hydrocarbons from the source rock at optimum maturity and is a measure of the quality of the source rock. For immature sediments, values of 0 to 2000 ppm of hydrocarbon characterise a poor source rock, 2000 to 6000 ppm fair, 6000 to 20 000 ppm good and above 20 000 ppm very good.

Pyrolysis techniques have in recent years provided a major advance in the assessment of source rock quality and generating potential. Hydrocarbon yields from immature source beds examined on-structure may be translated into actual oil productivity from the same beds in mature basinal, off-structure situations. Models relating maturity and kerogen type may be used to define original source rock quality grades which are of great value in mapping organic facies. Amorphous kerogen types, indistinguishable in microscopic preparations over a wide range of chemical properties, may be readily differentiated by pyrolysis. The problem of analysing bulk samples containing mixed kerogens has been largely overcome by the kerogen type/maturity model and anomalous results arising from the presence of caving contamination and drilling mud additives can usually be explained by inspection. High oxygen indices sometimes occur as a result of the presence of metastable carbonates and in such cases the sample is acid decarbonated and re-run.

#### Visual Examination of Kerogen Concentrates

All palynological preparations on which SCI determinations are made are also examined for kerogen type. Visual estimations of the relative abundance of the broad groups vitrinite, inertinite and sapropel are made on the total kerogen slide mount but reference is also made to the >20 $\mu$  sieved fraction to assist in identification. The scheme of identification is shown in Appendix Table 1. Full use is made of incident blue or UV light in distinguishing immature or early mature oil-prone kerogen from gas-prone kerogen.

#### Extract Analysis

The soluble organic materials present in rocks can be extracted with organic solvents, fractionated and analysed. The type and amount of material extracted depends largely upon the nature of the contained kerogen and its maturity, although the presence of migrant oil or drilling contamination may be the determining factors.

A maximum of 40g of crushed sample is extracted for a minimum of 12 hours in a Soxhlet apparatus using laboratory redistilled DCM. The solvent and the more volatile components (approximately up to  $n$ -C<sub>15</sub>) are lost by evaporation in an air flow and the resulting total extract is weighed, dissolved in hexane and separated into alkane (saturate) hydrocarbon, aromatic hydrocarbon, resene and asphaltene (polar) fractions by silica adsorption chromatography in the Iatroscan process.

Larger fractions, suitable for further analysis, are obtained by column chromatography. The extract is run through a short glass column packed with silica and alumina and eluted with hexane (to give the saturate fraction), (3:1 hexane: toluene mixture (to give the aromatic fraction) and methanol (to give the polar, or resene and asphaltene, fraction). A small proportion of non-eluted polar compounds usually remains on the column.

The data tabulated in reports comprise the following parameters:

Total extract - soluble organic matter, heavier than about  $n-C_{15+}$ , expressed as ppm of weight of rock.

Hydrocarbons - sum of alkane and aromatic hydrocarbons, expressed as ppm of weight of rock.

Extract % of organic carbon (EPOC) -  $\frac{\text{total extract ppm}}{\text{TOC} \times 100}$ ; the extractability.

Hydrocarbons mg/g of organic carbon - total hydrocarbons normalised to 1g of organic carbon.

Hydrocarbons % extract - total hydrocarbons as a proportion of total extract.

Alkanes % hydrocarbons - the proportion of alkanes (saturates) in the total hydrocarbons. The proportion of aromatics is (100 minus this value) expressed as a percentage.

The extractability of oil-prone sapropelic organic matter increases rapidly in the oil generation zone and diminishes to very low values in post mature sediments. Overall the extractability of sapropelic organic matter is greater than that of gas-prone humic organic matter for similar levels of maturity. Samples with extractabilities of greater than 20% generally contain migrant oil or are contaminated with mud additives.

As maturation proceeds in the oil generation zone the proportion of hydrocarbons in the total extract increases from less than 20% to a maximum in the most productive horizons of around 60%. This trend is reversed as the oil-condensate zone is entered. The relative proportions of alkanes to aromatics can be used as a check for low levels of contamination. Fractions of the extract, separated by column chromatography are retained for further analysis by gas chromatography or for stable carbon isotope determination.

#### Capillary Gas Chromatography of $C_{15+}$ Alkanes

A portion of the Soxhlet extract is eluted with hexane through a short silica column to yield the saturate hydrocarbon fraction. This fraction is evaporated in a stream of dry nitrogen at room temperature. A small portion of the fraction is then taken up in hexane and introduced into a 25 metre, wall-coated, open tubular glass capillary column coated with OV-1, or equivalent, mounted in a Carlo Erba gas chromatograph which is temperature programmed from 70°C to 270°C at 3°C/minute.

$C_{15+}$  chromatograms are inspected for the distributions of  $n$ -alkanes, and the presence and abundance of isoprenoids (particularly pristane and phytane), steranes and triterpanes and unresolved envelopes of naphthenic compounds. The ratios pristane:phytane and pristane: $n-C_{17}$  are calculated. Carbon Preference Index (CPI) values quoted are those as defined by Philippi as the ratio  $2C_{29}$  to  $(C_{28}+C_{30})$  unless otherwise stated. Chromatography may reveal information about the kerogen type of the source rock, its maturity and condition of deposition and, if migrant oil is present, whether this has been water-flushed or biodegraded. Contaminant drilling mud additives may be identified.

#### Capillary Gas Chromatography of Aromatic and Branched/Cyclic Alkanes

The aromatic portion of the Soxhlet extract is eluted from a short silica/alumina column by a hexane/toluene mixture. The dried fraction is taken up in DCM and introduced into a 25 metre, wall-coated, open tubular glass capillary column coated with OV-1, or equivalent, mounted in a Carlo Erba gas chromatograph which is temperature programmed from 70°C to 270°C at 3°C/minute.

Branched chain alkanes are separated from normal alkanes by urea adduction and treated as for total alkanes.

### Gas Chromatography-Mass Spectrometry

Mass spectrometry is a technique in which molecules are bombarded with high energy electrons causing ionisation and fragmentation of the molecules into ions of varying mass(m) and charge(z). The way in which a molecule fragments into ions of various m/z value is known as its fragmentation pattern, or mass spectrum and is unique. When linked to a gas chromatograph the mass spectrometer can be used in two different modes:

1. Full Scan Mode: A mass spectrum is obtained of each peak eluting from the gas chromatograph and a structural identification of the compound producing that peak can be made.
2. Multiple or Single Ion Monitoring Mode: The mass spectrometer is tuned to certain m/z values to detect whether a compound, eluting from the gas chromatograph, fragments to give an ion at that value. Certain fragmentations are indicative of specific compound types and the most commonly monitored fragment ions used in petroleum geochemistry are those with m/z values of 191, 217 and 259 which are the principal fragment ions obtained from groups of alkanes known as triterpanes, regular steranes and rearranged steranes respectively. These are compounds containing 27 to 35 carbon atoms arranged in a polycyclic, normally 4 or 5 ring, structure, occurring in the  $n-C_{26}$  to  $n-C_{35}$  region of a gas chromatogram. The basic molecular skeletons of these compounds are very similar to those of the original organic matter deposited in the sediment and so these 191, 217 and 259 distribution plots, known as mass fragmentograms or mass chromatograms, form a pattern characteristic of the source material. This technique of "fingerprinting" is also one of the more exact methods of correlating an oil to its source, or to another oil.

### Carbon Isotope ( $^{13}C/^{12}C$ ) Ratio Analysis

Carbon has two stable isotopes, the more abundant  $^{12}C$  isotope and the heavier  $^{13}C$  isotope, which in nature forms about 1% of carbon. Deviations from the  $^{13}C/^{12}C$  ratio are extremely small and carbon isotope ratios, as measured by mass spectrometry, are expressed as deviations from a standard, the Pee Dee Belemnite carbonate (PDB standard) in parts per thousand (parts per mil; ‰). Positive deviations indicate  $^{13}C$  enrichment and conversely, negative deviations indicate  $^{13}C$  impoverishment.

While the carbon isotope ratios of oils and rock extracts can range from -20 to -32 ‰ depending on the source organic matter type, the difference between a specific oil and its source is small. Measurements are usually made on the  $C_{15+}$  alkane and aromatic hydrocarbon fractions separately and there should be no more than 1 ‰ difference between the oil and its source for either fraction. If there is any doubt that the source rock extracts are not indigenous to the source rock kerogen, the carbon isotope ratio of the extracted source rock kerogen can be measured.

### Pyrolysis-Gas Chromatography

The hydrocarbon pyrolysate derived from thermal, anhydrous cracking of kerogen is analysed by capillary gas chromatography. A few mg of rock, kerogen or asphaltene is heated to 600°C for 20 seconds in the injector of a gas chromatograph. The chromatograph oven is kept at -30°C during pyrolysis and then raised to 300°C at a programmed rate of 7.5°C/minute. Chromatograms produced this way are often very different from those of source rock extracts or oils in that branched and cyclic isomers are generated freely giving numerous, closely spaced peaks, along with unsaturated, alkene (olefin) hydrocarbons. The "doublet" peaks often observed in these chromatograms comprise alkene-alkane pairs, the first eluting, and usually smaller peak, being the alkene. The chromatograms range from  $C_1$  to  $C_{30}$  or above and although variable, are broadly characteristic of source rock type. Gas-prone kerogen cracks to give a more limited molecular weight range of products, concentrated towards the light ends, whereas oil-prone kerogen gives more prominent alkene-alkane doublets in the  $C_{12}$  to  $C_{30}$  region. The largest peak from both types is usually methane.

### Elemental Analysis

Total (unsieved) kerogen is prepared as described in Section 1. The dried material is combusted in oxygen in an elemental analyser and the oxides of carbon, hydrogen, nitrogen and sulphur are measured. The unburnt residue is the ash content. Oxygen is usually calculated by difference but can be determined separately if required. Results are quoted as percentage weights of C, H, O, N, S and Ash with the atomic ratio H/C and O/C calculated and plotted on the standard van Krevelen diagram. The relative amounts of C, H and O present in organic matter are dependent on both source and maturity. At known maturity levels, some measure of source quality may be determined. Limitations of the method in source rock assessment involve the difficulty of obtaining pure kerogen (in particular, free from pyrite) and the lack of a simple, direct determination of oxygen content.

#### 4. Oil Analysis

RRI laboratories offer a wide range of oil analyses both for geochemical purposes and industrial use. Physical property determinations are based mainly on IP methods and are available for lubricating oils, fuels and greases as well as crudes. Frequently measured properties of crude oils presented in geochemistry reports include: API gravity, pour point, viscosity and contents of water, sulphur, wax, asphaltene, nickel, vanadium and other metals. Chemical analysis of oils involves the following:

Whole oil gas chromatography - using split syringe injection and a temperature programme from -20°C or -30°C up to 270°C at 4°C/minute.

Associated gas - if oil has high gas/oil ratio.

Gasoline analysis - as for gasolines in rock samples but a weighed quantity of oil is used.

Topping of the oil - this is equivalent to the removal of the fraction boiling below about 210°C and gives a more standardised product for comparison of gas chromatograms of the C<sub>15+</sub> fraction.

Column chromatography and gas chromatography - as for solvent extracts. Analysis is carried out on topped oil.

#### 5. Gas Analysis

The hydrocarbon gases, C<sub>1</sub> to C<sub>4</sub>, may be collected from the airspace of sealed canned samples or may be received from well-site tests in a special sealed gas cylinder (gas mouse). Chromatographic separation of the C<sub>1</sub> to C<sub>4</sub> gases is effected as described under airspace gas analysis. In addition, the separated gas components may be analysed for stable carbon and hydrogen isotope composition which may provide valuable clues to the origin of the gas.

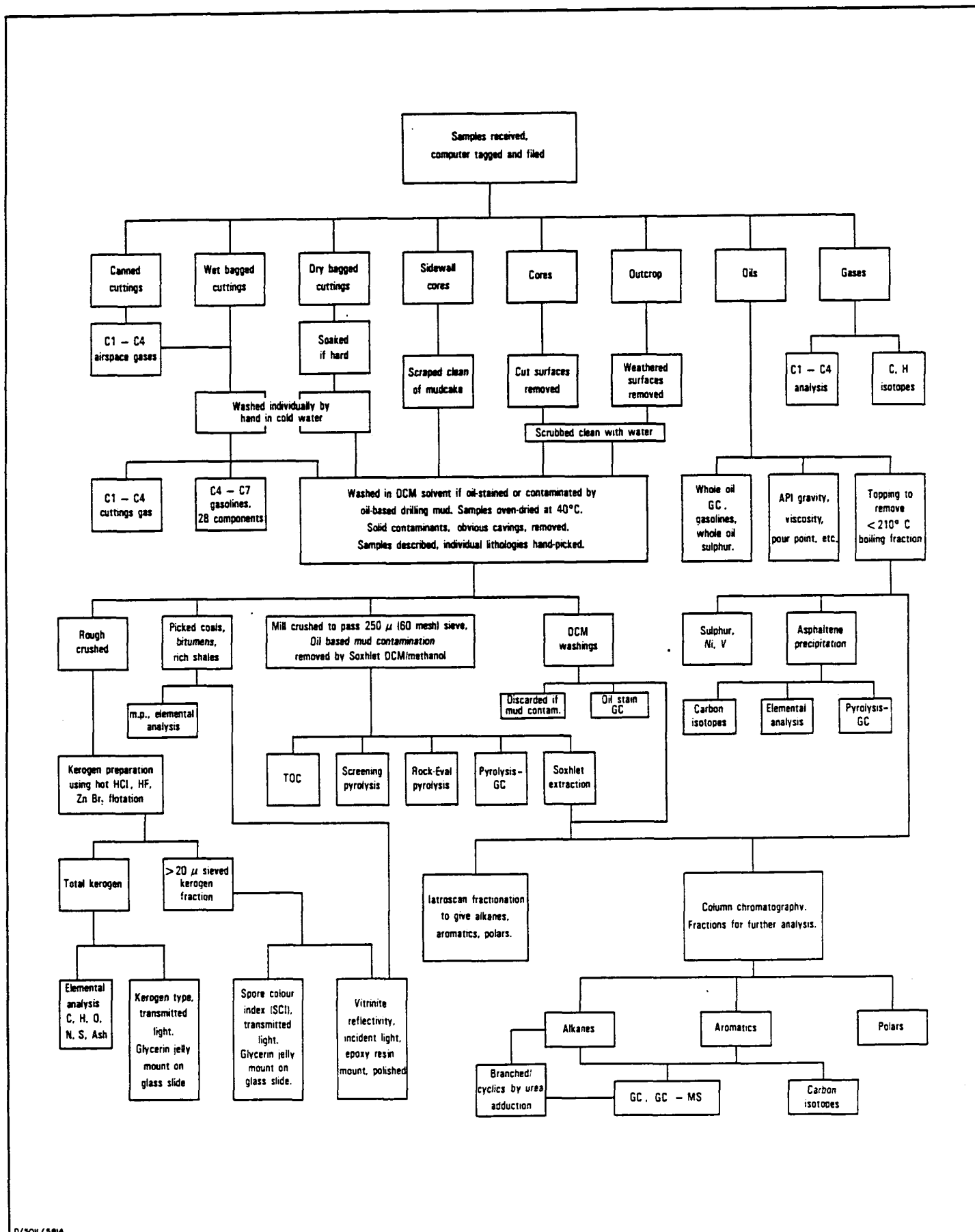
#### 6. Solid Bitumen Analysis

In some oil fields, problems are encountered where bitumen developments form continuous or patchy layers within reservoirs, dividing the pay zones and acting as barriers to natural fluid movement or inhibiting enhanced oil recovery techniques. Integrated geochemical and sedimentological studies aim to produce geological models capable of predicting the occurrence of bitumen layers and their likely thickness and ability to act as permeability barriers. Of further concern are the past or present relationships between the bitumen and reservoir oil, their source rocks and the timing of bitumen formation.

Analysis schemes involve screening of samples by assessing the amount of bitumen in polished core pieces using reflected light microscopy, followed by solvent extraction of control samples to estimate the proportion of solvent soluble bitumen. Different phases of bitumen formation are differentiated by reflectance measurement as described for vitrinite reflectance measurement. Soluble extracts are fractionated to give alkane, aromatics, asphaltene and resene components. Separated bitumens may be subjected to elemental analysis.



# FLOW CHART FOR GEOCHEMICAL ANALYSIS



D/50H/5814

APPENDIX FIGURE 1

Kerogen Typing Scheme for Transmitted White and Incident Blue/U.V. Light

General Properties	RRI Report Data Tables	Type *
Sapropelic (Oil-prone gas-prone at high maturity)	Algal Sapropel	Type I
	Waxy Sapropel	Type II
Humic (Gas-prone)	Vitrinite	Type IIIA
	Inertinite	Type IIIB

Amorphous		Structured	
Non-Fluorescent	Fluorescent	Non-fluorescent	Fluorescent
Type I/II at high maturity (SCI >7.5)	Type I Sapropel Type II (degraded spores) Soft bitumens	Vitrinite (Type IIIA) brown/black, woody tissue	Cuticle Spores Pollen Dinocysts (Type II)
Type IIIA/B			
Oil residues (bitumens) Mineral (undigested) Grease contamination Mud additives		Inertinite (Type IIIB) very dark brown/black, woody tissue	Resinite Algae (Tasmanites, Botryococcus etc.)  (Type I)
		Solid bitumen - brown/ black (oil residue) often with crystal imprints	
		Microforaminifera, chitinozoa etc. (Not usually important)	
		Spores, cuticle etc. at high maturity levels	
		Mud Additives - walnut etc.	

\* Types I, II, III approximately sensu Tissot et al but Type III subdivided into IIIA (vitrinite) and IIIB (inertinite)

APPENDIX TABLE 1

APPENDIX 3.  
"Rock Eval" Pyrograms

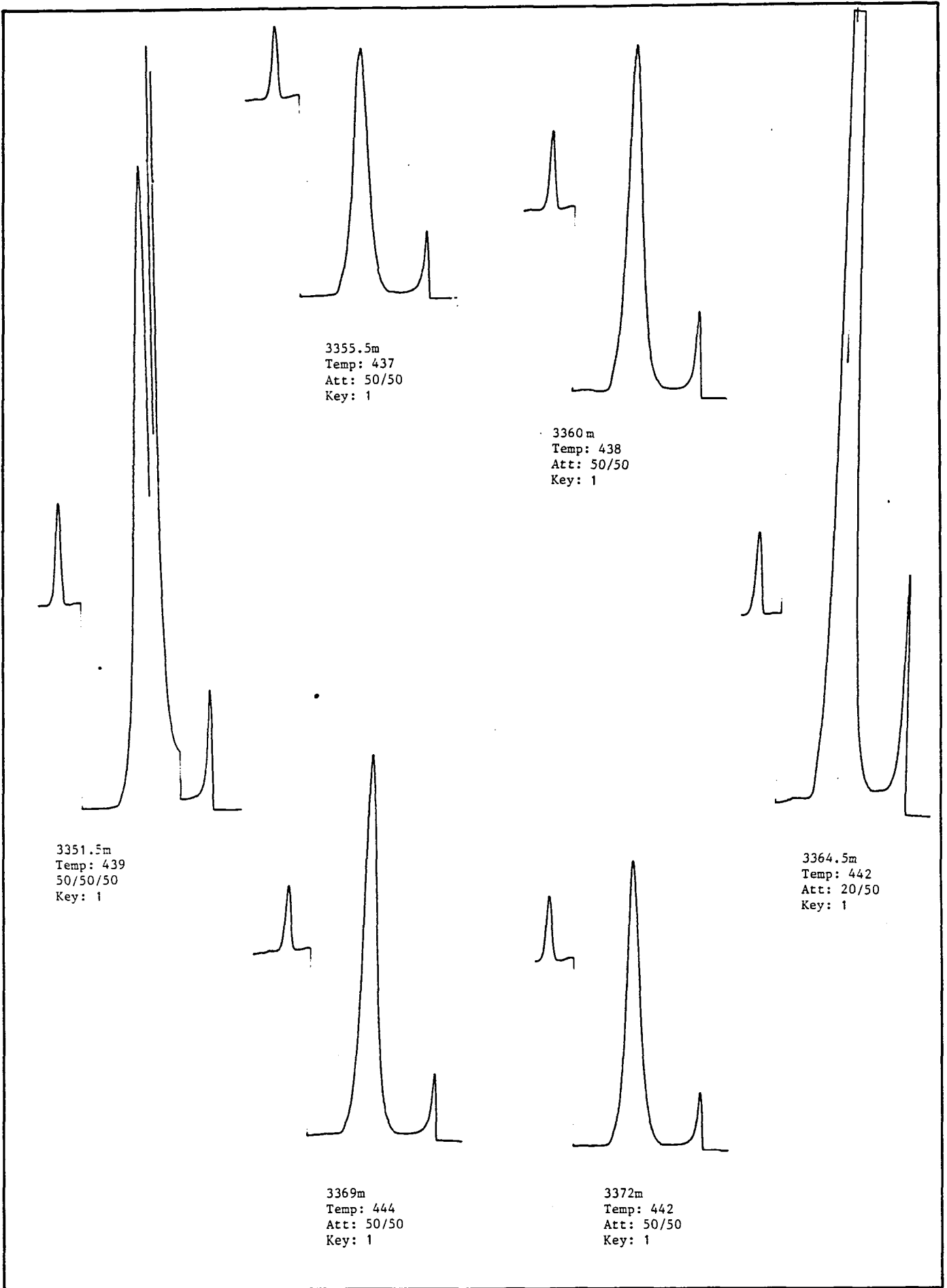
(9 sheets)

"ROCK-EVAL" PYROGRAMS

COMPANY: NORSKE SHELL

WELL: 30/11-4

LOCATION: NORWEGIAN NORTH SEA



KEY TO ABBREVIATIONS

A. Pyrogram of sample:- 1. As Received

2. After Extraction

3. After Decarbonation

4. After Extraction and Decarbonation

B. Att.- Instrument Attenuation

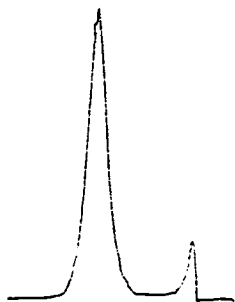
C. Temp.- "Rock-Eval" T Max.

"ROCK-EVAL" PYROGRAMS

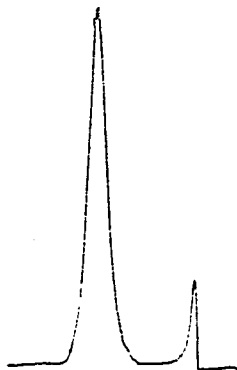
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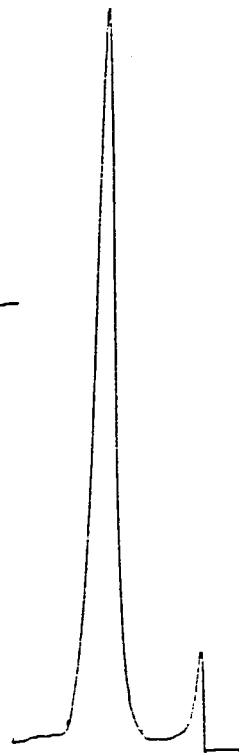
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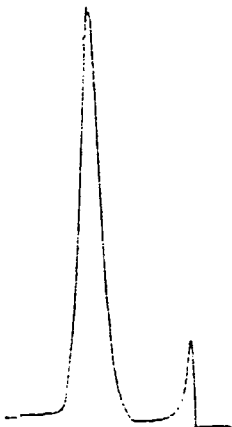
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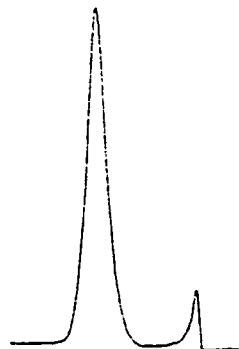
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Key: 1



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Key: 1



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KEY TO ABBREVIATIONS

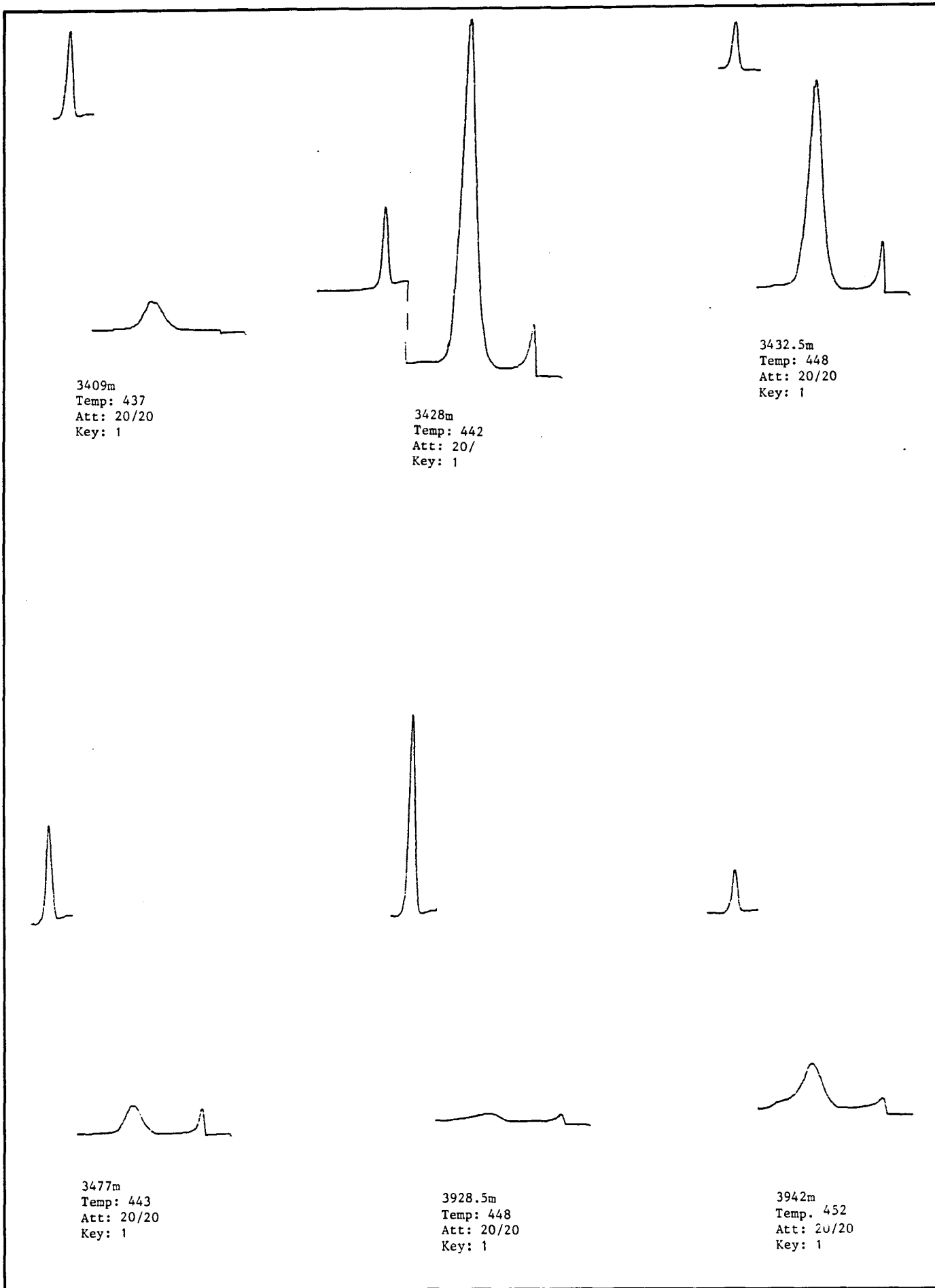
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|-------------------------|---------------------------------------|----------------------------------|
| A. Pyrogram of sample:- | 1. As Received                        | B. Att.:- Instrument Attenuation |
|                         | 2. After Extraction                   | C. Temp.:- "Rock-Eval" T Max.    |
|                         | 3. After Decarbonation                |                                  |
|                         | 4. After Extraction and Decarbonation |                                  |

"ROCK-EVAL" PYROGRAMS

COMPANY: NORSKE SHELL

WELL: 30/11-4

LOCATION: NORWEGIAN NORTH SEA



KEY TO ABBREVIATIONS

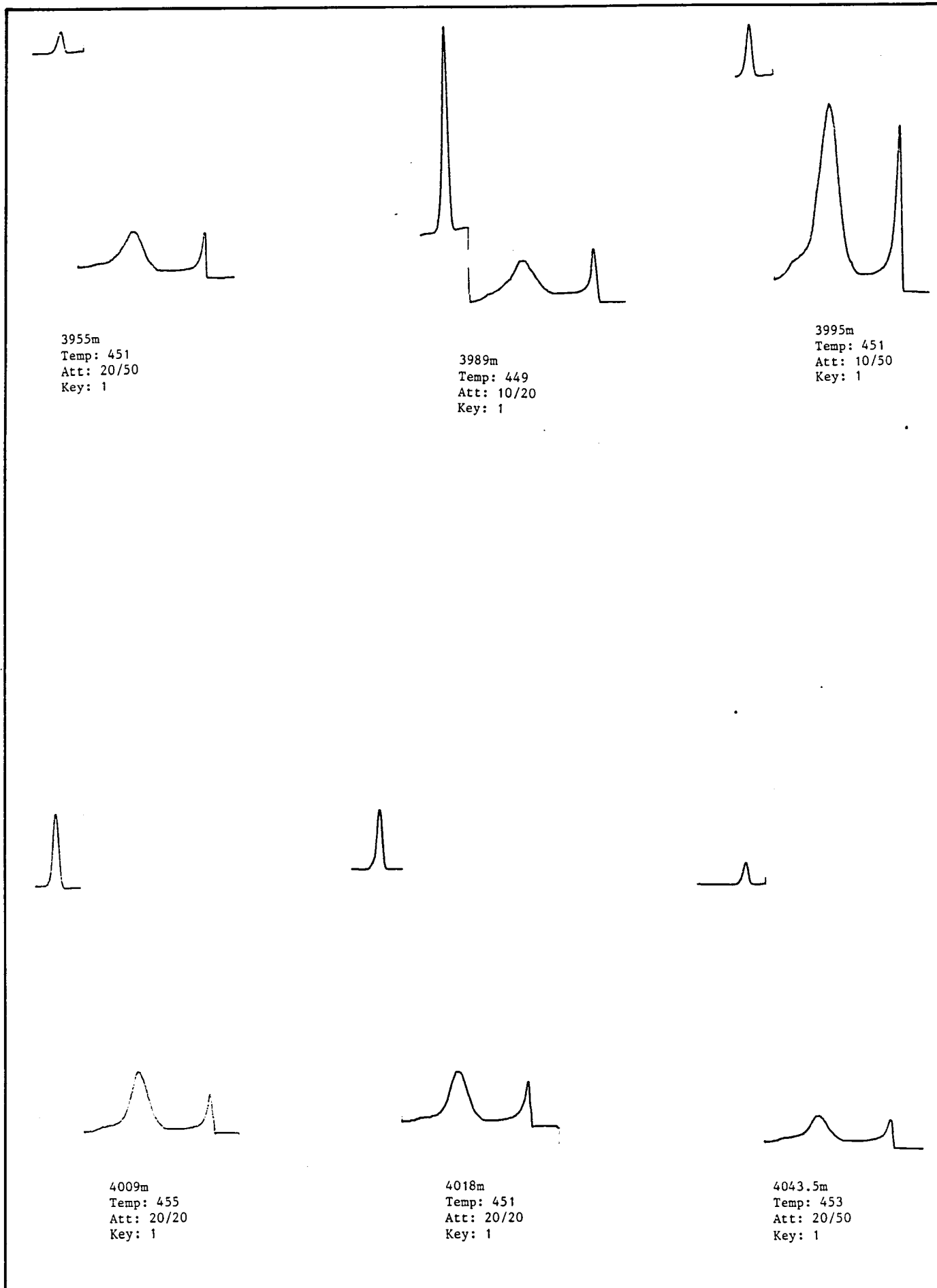
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"ROCK-EVAL" PYROGRAMS

COMPANY: NORSKE SHELL

WELL: 30/11-4

LOCATION: NORWEGIAN NORTH SEA



KEY TO ABBREVIATIONS

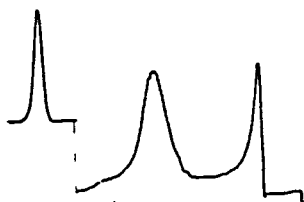
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  3. After Decarbonation
  4. After Extraction and Decarbonation
- B. Att.-: Instrument Attenuation  
C. Temp.-: "Rock-Eval" T Max.

## "ROCK-EVAL" PYROGRAMS

COMPANY: NORSKE SHELL

WELL: 30/11-4

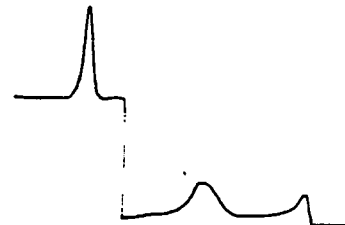
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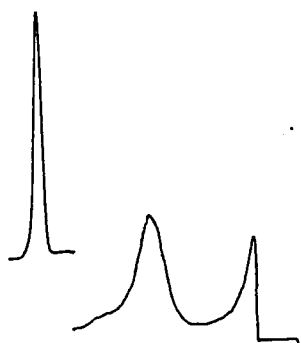
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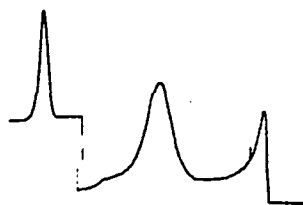
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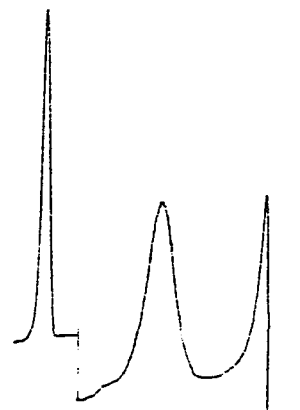
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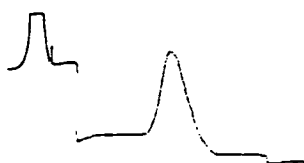
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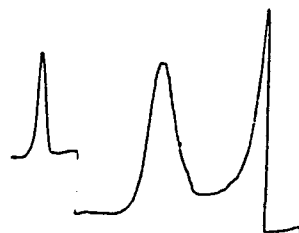
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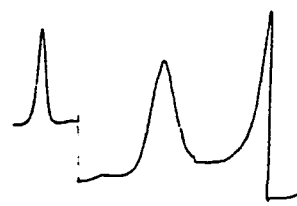
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Key: 1



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## KEY TO ABBREVIATIONS

A. Pyrogram of sample: 1. As Received

2. After Extraction

3. After Decarbonation

4. After Extraction and Decarbonation

B. Att.: Instrument Attenuation

C. Temp.: "Rock-Eval" T Max.



"ROCK-EVAL" PYROGRAMS

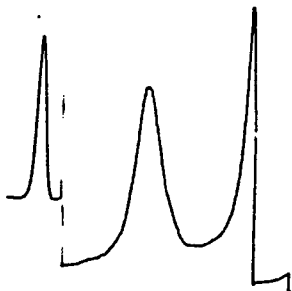
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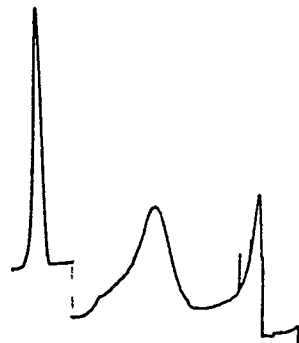
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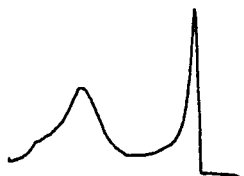
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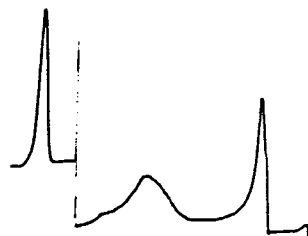
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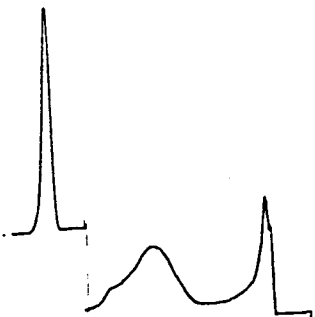
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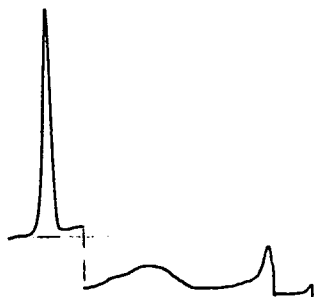
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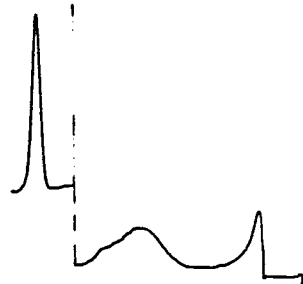
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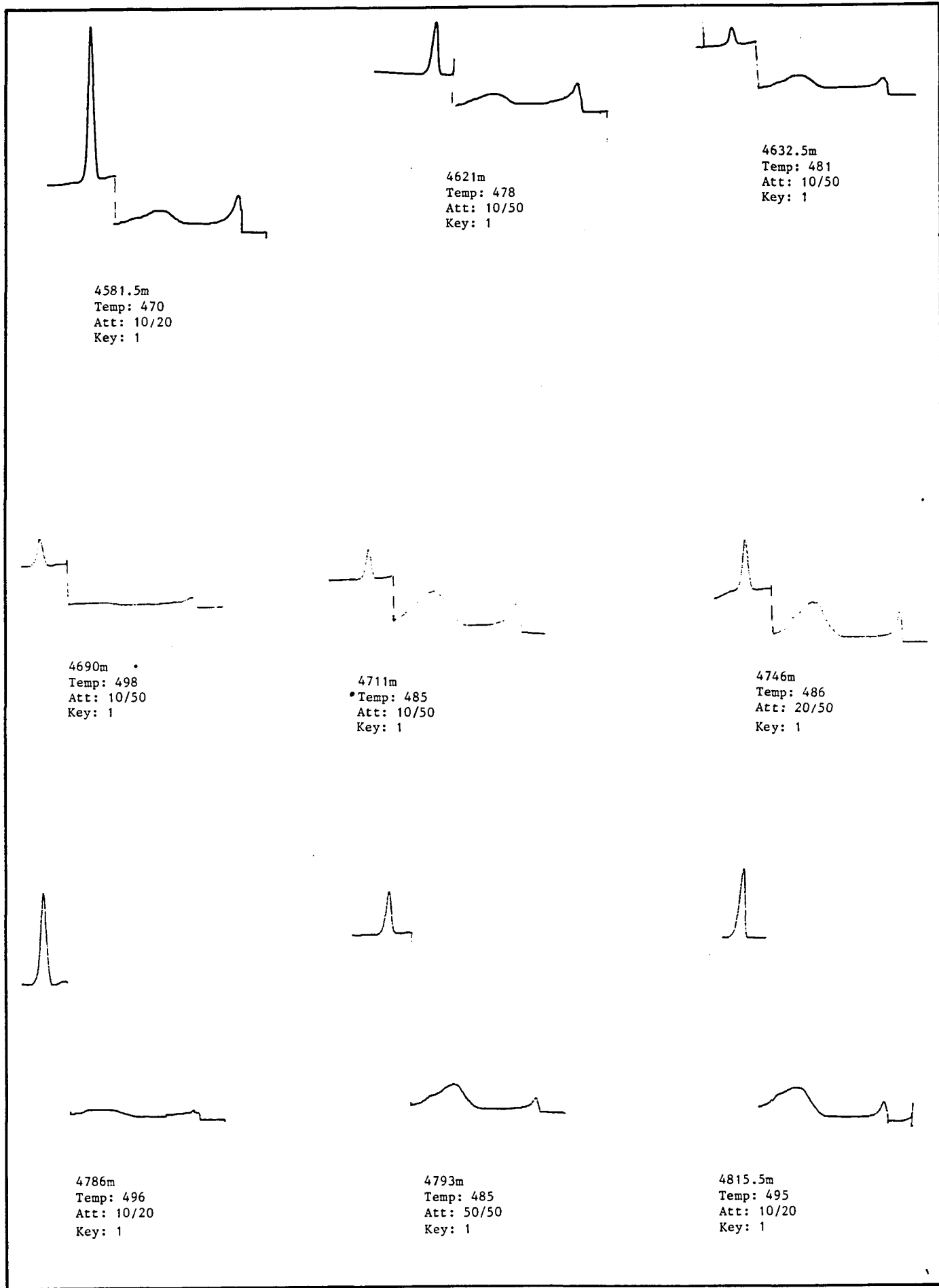
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|---|--|
| <p>A. Pyrogram of sample:-</p> <ol style="list-style-type: none"> <li>1. As Received</li> <li>2. After Extraction</li> <li>3. After Decarbonation</li> <li>4. After Extraction and Decarbonation</li> </ol> | <p>B. Att.: Instrument Attenuation</p> <p>C. Temp.: "Rock-Eval" T Max.</p> |
|---|--|

"ROCK-EVAL" PYROGRAMS

COMPANY: NORSKE SHELL

WELL: 30/11-4

LOCATION: NORWEGIAN NORTH SEA



KEY TO ABBREVIATIONS

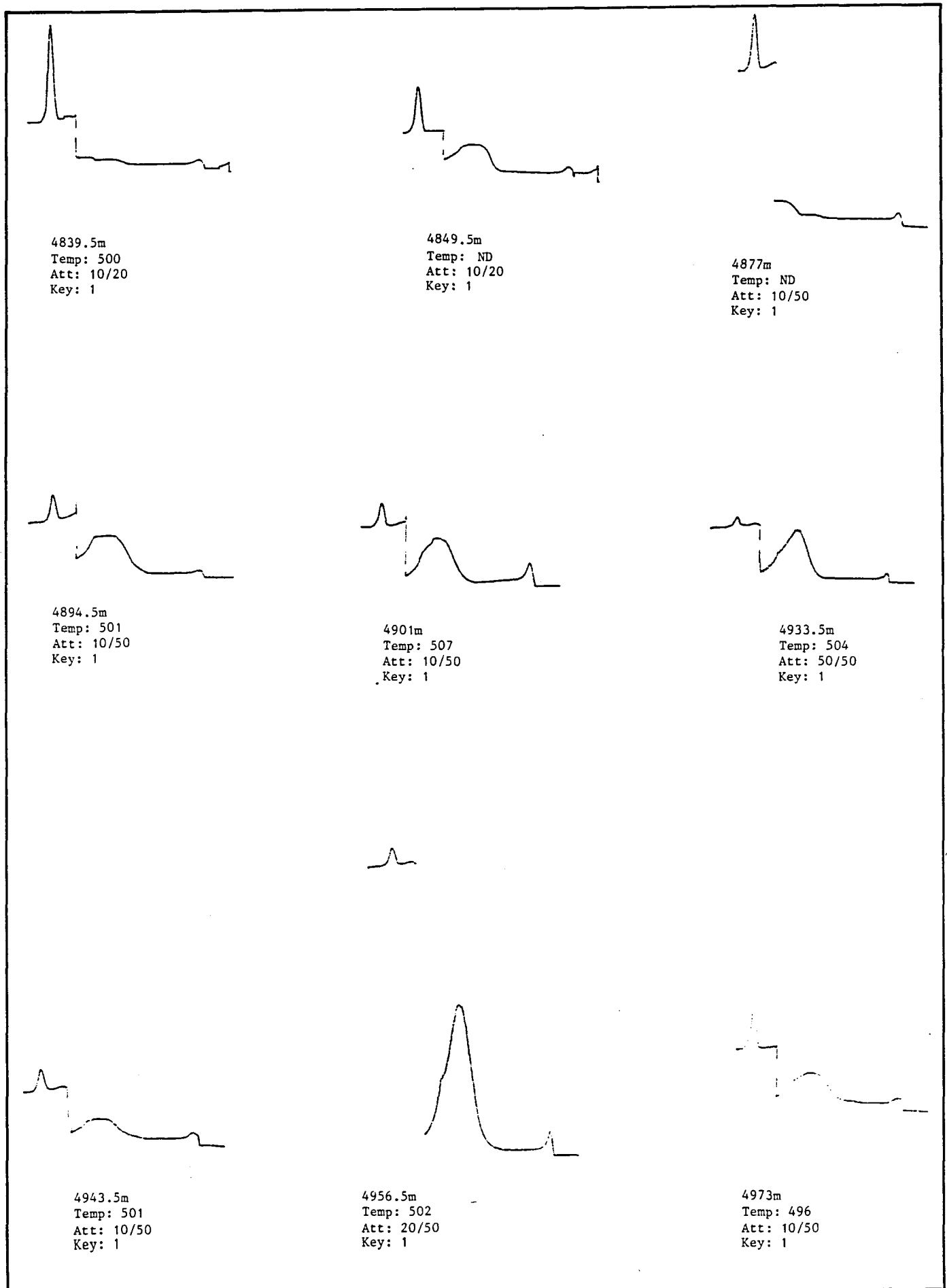
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|---|--|
| <p>A. Pyrogram of sample:-</p> <ol style="list-style-type: none"> <li>1. As Received</li> <li>2. After Extraction</li> <li>3. After Decarbonation</li> <li>4. After Extraction and Decarbonation</li> </ol> | <p>B. Att.- Instrument Attenuation</p> <p>C. Temp.- "Rock-Eval" T Max.</p> |
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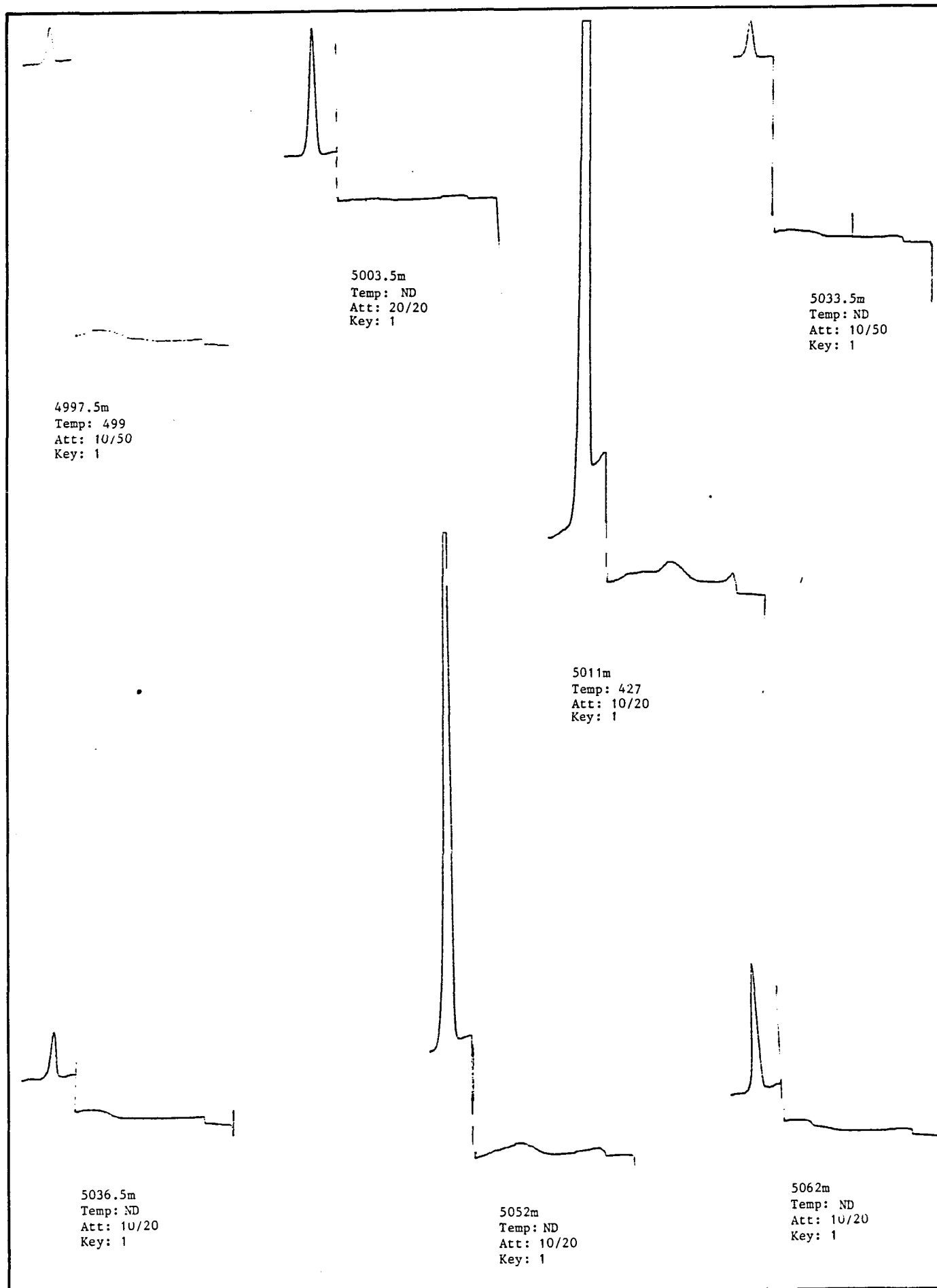
- |   |  |
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