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CONFIDENTIAL

AN ADSORPTION METHOD FOR THE EXTRACTION OF KRYPTON AND XENON

Part I

V. G. Fastovskiy,
Lab of Rare Gases,
All-Union Electro-Tech Inst
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The rarest components of the air -- krypton and xenon -- have in recent years acquired tremendous practical importance, due to their high atomic weight, inertness, and low heat conductivity.

The remarkable growth of the technology of gas liquifaction, on which the hydrogen and nitrogen industries are principally based, and the liquifaction of coking gas have created the prerequisites for obtaining krypton (Kr) and xenon (X) in significant quantities, and this has been widely reflected in practices used abroad.

In the process of the extraction of Kr and X from air (NOTE: A description of this process is outside the bounds of the present report) oxygen is obtained which contains Kr and X in amounts of 0.1 to 0.3% and together with insignificant admixtures of argon; this oxygen, which we refer to in the ensuing account as a concentrate, contains 1000-3000 times as much Kr and X as the initial gas, i.e., air.

The reprocessing of this concentrate into pure Kr and X represents a serious technological problem; it is conceivable to use the method of burning hydrogen with the concentrate and subsequently separating krypton and xenon from the combustion products (NOTE: We are developing a method of this type. It seems possible to employ the method of subsequent distillation of the concentrate with the purpose of obtaining a liquid phase enriched in Kr and X).

The present investigation is pursuing a strictly practical aim: to ascertain the possibilities and the optimum conditions for the reprocessing of

the concentrate by the adsorption method with the goal of obtaining these gases (Kr and X) in the pure form.

In principle it is fully possible to pass the cold concentrate directly from the distillation or washing column through an apparatus filled with some kind of adsorbent (activated carbon, silica gel) for the purpose of effecting retention by adsorption of the high-boiling, heavy components krypton and xenon (NOTE: In the rest of this account we will refer only to krypton, although all conclusions relating to krypton are definitely applicable to xenon). A similar method for the separation of gaseous mixtures by the method of fractional adsorption is based on the well-known relation which can be expressed in the following manner: the more the components of the gaseous mixture to be separated differ from each other as far as their physical properties (boiling point, critical temperature, and atomic weight) are concerned, the easier and simpler it is to separate such a mixture. This relation, true for any method of separating a gaseous mixture, is also completely applicable to the adsorption method.

Large-scale and extremely interesting investigations on the adsorption of rare gases by activated carbon have been carried out by K. Peters and K. Weil, whose works we shall submit to examination below. We will note that K. Peters and W. Lomar [1] showed the method of fractional adsorption to be capable of effecting the separation of isotopes.

It is well known that for the determination of the optimum technological conditions of separation of any gaseous mixture it is necessary to have data on the adsorption value of the individual components of the mixture under consideration. In accordance with the composition of the gaseous mixture (concentrate) which was of interest to us, we conducted adsorption measurements with silica gel on O_2, Ar, and Kr.

In Figure 1 is shown the diagram of the apparatus for investigation of the adsorption of gases.

The gas being studied is located in the glass cylinders A or A_1. The gas from the cylinders goes into the graduated burette B, and its pressure

is recorded by the manometer C. Then, with the burette being used as a mercury pump of the Toepler type, the gas is compressed into the adsorber E which is placed in a Dewar flask containing a cooling mixture of liquid nitrogen and alcohol. The pressure in the adsorber which is established on the completion of the adsorption process is recorded by the manometer C_1. Small pressures on the adsorbent are recorded by the MacLeod manometer D.

Prior to the beginning of the experiments the whole apparatus is carefully evacuated, and the silica gel is warmed up and subsequently evacuated. The volumes of the input line and the inactive space in the adsorber E were carefully determined by filling the separate parts of the apparatus with helium.

We obtained pure oxygen by heating $KMnO_4$. Argon was obtained by distilling the argon-nitrogen mixture [2] and subsequently adsorbing the remaining nitrogen with powdered metallic calcium. The purity of the oxygen and nitrogen was determined by weighing these gases with a balance of the Stock and Ritter type [3], the density of the oxygen (relative to air) being equal to 1.1, and the density of argon = 1.376 (the density of pure argon is 1.38).

It was a more complicated matter to obtain pure krypton; the Kr-X mixture which we obtained was subjected to partial distillation for the purpose of decreasing the content of xenon in the gas. The krypton taken for subsequent investigations possessed a density of 2.85, which corresponds to the following composition of the gas: 98% Kr and 2% X (the density of pure krypton relative to air is 2.82).

We conducted measurements at temperatures from -55 to -120°. The temperature during the process of taking an isotherm varied within limits of 1-2°. Temperature measurements were taken with a copper-constantan thermocouple set directly in the adsorber E. The weighed in portion of adsorbent was 2.8 g.

In Tables 1, 2, and 3 are shown the results of our measurements.

Here p is the equilibrium pressure in the adsorber and a is the value of adsorption in cu cm. When there is a simple linear dependence between p and a, the value a/p (column 3) must remain constant.

Thus Lambert and Peel, having studied the adsorption of nitrogen and oxygen by silica gel at 0°, showed that for this temperature down to 700-800 mm Hg the relation a/p remained constant. Unfortunately, these authors did not carry out measurements at low pressures (their measurements begin approximately with 100 mm Hg), which does not permit conclusions to be drawn about the character of the whole isotherm (0°)

The results of our measurements are presented graphically: the adsorption isotherms (-55°) for Kr, Ar, and O_2 in Figure 2; and in Figures 3 and 4 the corresponding ones for -93° and -120°.

At a temperature of -55°, the adsorption of Ar and O_2 has a practically linear character, but this by no means applies in the case of Kr. At lower temperatures this relation becomes extremely complicated for all the components of interest to us (Ar, O_2, and Kr). In a definite pressure range (to 200-300 mm Hg) this relationship is satisfactorily expressed by Freidlich's parabolic equation:

$$a = \alpha \cdot p^{1/n} \qquad (1)$$

where a is the adsorbed quantity of gas, p is the pressure over the adsorbent, and α and 1/n are constants. The dependence between a and p in the coordinate system lga/lgp has a linear character and permits an easy graphic determination of the constants α and 1/n.

Let us turn to a consideration of the basic problem of the present investigation: to a consideration of the determination of the optimum conditions for separation of the gaseous mixture (concentrate) in which we are interested.

The basic factors of this process for the separation by adsorption of the gaseous mixture are as follows: 1) the method for reprocessing the gaseous mixture (fractional adsorption or fractional desorption); 2) temperature; 3) pressure; 4) quantity of adsorbent; 5) mutual effect of the components of the gaseous mixture as reflected on the adsorption process (it is

known that the presence of one component has an effect on the amount of adsorption of another component); 6. adsorbent (activated carbon, silica gel).

Let us attempt to elucidate the influence of each of the factors enumerated. Fractional adsorption is in essence the passing of a gaseous mixture continuously through an adsorbent and subsequently desorbing the adsorbed portion of the gas. From the engineering and production point of view this method is extremely attractive, since with a suitable system for switching over the lines it is possible to produce a continuous flow of gas with an insignificant quantity of adsorbent. However, such a process of dynamic adsorption can lead to a considerable loss of krypton, for the higher the boiling point of a component and the lower the temperature of the adsorbent, the longer the time necessary to reach adsorption equilibrium.

In the investigation of the adsorption of rare gases [5] we had the opportunity to satisfy ourselves that in a given temperature range the adsorption equilibrium for helium and neon is reached very quickly, while for heavy rare gases the process continues for a long time. This circumstance in the use of dynamic fractional adsorption requires a very low velocity of gas flow, and this is negatively reflected in the dimensions of the adsorption apparatus.

There is no doubt that from the point of view of reducing losses of krypton it is preferable to use the method of fractional desorption: the given adsorber is saturated up to a certain pressure with the concentrate, and then the process of fractional desorption is accomplished with a vacuum pump. Such a method, when the correct thermal conditions are chosen, not only permits all the oxygen and argon to be separated from the adsorbent, but also permits the residual gas (Kr and X) to be obtained in the pure form, without substantial losses.

The practical question arises as the whether the process of fractional desorption should be carried out by the gradual heating of the adsorbent or by the evacuation of the adsorbent at a constant temperature. The experience of our work and the investigations of K. Peters and K. Wall [6] show

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The practical question arises as the whether the process of fractional desorption should be carried out by the gradual heating of the adsorbent or by the evacuation of the adsorbent at a constant temperature. The experience of our work and the investigations of K. Peters and K. Well [6] show

that in order to effectively separate the gaseous mixture and obtain krypton and xenon in the pure form the adsorber should be evacuated at the selected adsorption pressure (see below) down to a limiting low pressure and the residual gas separated by heating the adsorbent.

The temperature factor is of decisive importance for effective separation of the gaseous mixture by the adsorption method. Investigations on adsorption of gases shows that within a specified temperature interval the adsorption takes place reversibly (not in the thermodynamic sense of the word), i.e., the curves of adsorption and desorption fully coincide. We conducted similar experiments with helium and neon at the temperature of liquid nitrogen; for helium the adsorption and desorption curves were fully coincident, but for neon they showed an insignificant divergence.

The fact that the process takes such a course deprives us of the opportunity to separate the individual fractions in their pure form in the desorption [7]. However, as the researches of K. Peters and K. Well [6] showed, there is for each gas a so-called critical temperature of adsorption; below this temperature the adsorption and desorption processes do not proceed reversibly. This critical temperature of adsorption has a physical significance which differs little from the ordinary critical temperature of a given gas.

The facts stated above permit a very important practical conclusion to be reached: if the process of adsorption of a gaseous mixture is carried out at a temperature lower than the critical temperature of one gas and higher than that of another, then in the subsequent desorption pure fractions will be capable of being separated in a single operation.

Thus K. Peters [8] showed that at a temperature of -225° (the critical temperature of neon is -228.8°) a neon-helium mixture could be completely separated without the formation of intermediate fractions containing neon and helium in various proportions.

The considerations cited have a direct relation to the mixture we are interested in (the concentrate consisting of O_2, Ar, and Kr). In the given case we dispose of a sizeable temperature interval (the critical temperature

of krypton is -63.81°, while those of oxygen and argon are respectively -118.8 and -122.4°) which greatly facilitates the separation of krypton (and to a still greater extent, of xenon) in a pure state. In the light of what is stated above it is clear that the process of adsorption of the mixture with which we are concerned must be carried out in the temperature interval located below the critical temperature of krypton and somewhat above the critical temperatures of O_2 and Ar. It must be noted, however, that unlike the ordinary critical temperature the critical temperature of adsorption of a given gas is by no means a fixed, unchanging quantity, but depends on a series of complex factors: the character of the surface of the adsorbent, the method of its treatment, and the nature of the other components of the gaseous mixture.

Thus K. Peters [8] introduces an interesting example. The investigator referred to succeeded in desorbing at 0° the whole quantity of adsorbed propane, while in the following experiment there was adsorbed the same quantity of propane as well as a certain quantity of butane. It appeared that in the subsequent desorption the whole quantity of propane was separated not at 0°, but at -10°, and even at -20°. Here the more volatile component (propane) was displaced from the adsorption surface by the less volatile, and this naturally resulted in a shift of the critical temperature of adsorption of propane.

In the case with which we are concerned it should be expected that the adsorbed krypton would contribute to the displacement of Ar and O_2 from the adsorption surface and would thereby facilitate the desorption of these gases.

In the separation of a gaseous mixture by the fractional condensation or fractional evaporation methods the vapor tensions of the components of the mixture are of basic importance. By analogy, when the gaseous mixture is being separated by adsorption, the pressures over the adsorbent (the unadsorbed phase) which correspond to a given value of adsorption (isostere) should be compared for a series of temperatures and the components of interest to us.

In Table 4 are cited the respective data from our experiments for adsorption values equal to 2, 4, 6, 9, and 12 cu cm of gas.

It is evident that the greater the ratio of the pressures in the unadsorbed phase of the components which are of interest to us, the more

favorable are the conditions for fractional desorption. This ratio is indicated by us in the last column of Table 4.

Let us note that similar calculations were carried out by K. Peters and K. Well [6] for the case of the adsorption of Kr, X, and Ar by activated carbon; in principle an analogous direction of the process was shown.

As is evident from the data cited, lowering of the temperature and reduction of the magnitude of adsorption per unit of adsorbent improve the conditions for the separation of the gaseous mixture, but in this case the reciprocal influence of the components of the mixture on the adsorption process was not taken into account. However, in adapting the process for production one should not proceed along the line of the maximum lowering of temperature and excessive increasing of the quantity of adsorbent, for this will lead to an extreme retardation of the desorption process, to excessive loading of the adsorbent with adsorbed O_2 and Ar, and to a considerable increase of the inactive space in the apparatus, all of which is undesirable for operation with such valuable gases as krypton and xenon.

We assume that the optimum temperature for the process of adsorption and subsequent desorption must be taken as -120 to -130°.

The influence of pressure on the ratio of the components in the adsorbed phase is presented in Table 5, which we compiled from experimental data.

Table 5 was made up for a temperature of -100°. As is evident from the data cited, lowering of the temperature increases the relative quantity of krypton in the adsorbed phase; it must be expected that when the mixture is adsorbed the ratio of the components in the adsorbed phase gives more favorable indices for krypton with an increase of pressure.

In Figure 5 are shown the isobars of adsorption of Kr, Ar, and O_2. From these curves, besides the temperature dependence of the amount of adsorption, it is possible to obtain data on the ratio of components in the adsorbed phase for a given isobar (800, 500, 200, 50 mm Hg) and for a given temperature.

Comparing the data of K. Peters and K. Well [6] with the data from our measurements, it is easy to reach the conclusion that in order to adopt for production the process of treating the concentrate preference must be given to activated carbon, and not to silica gel. The use of activated carbon permits the volume of the apparatus to be reduced, improves the ratio of components in the adsorption phase, and prevents to a greater extent than does silica gel the loss of krypton during the desorption of the gas.

It has been shown that when silica gel is used at the same temperature of adsorption, the process of adsorption and desorption exhibits a greater degree of reversibility than with activated carbon.

The effect of the adsorption of the gaseous mixture on the adsorption value of each component and on the desorption process has been much less studied.

Thus Damkohler [9] investigated the adsorption of an Ar-N_2 mixture on silica gel at temperatures of 89.5, 111, and 163° K (100-700 mm Hg), as a result of which he came to the conclusion that in the first adsorption layer preferential adsorption of N_2 took place, but that in subsequent layers Ar was adsorbed. Depending on the temperature conditions, the partial pressures of the components in the gaseous phase, and the common pressure on the adsorbent, an alternate shifting of the Ar and N_2 ratio in the adsorption phase takes place.

Frolich and White [10] studied the adsorption of a CH_4-H_2 mixture. It seemed that in this case a considerable adsorption of H_2 took place at insignificant pressures (1-2 atm), but that at high pressures (60-80 atm) hydrogen was almost absent in the adsorbed phase.

E. Markman and A. Benton [11] studied the adsorption of CO and O_2 on silica gel (0 and 100°), with the result that they disclosed the preferential adsorption of the highly volatile CO component. We will mention the investigations of R. Lorentz and E. Wiedbrauck [12], A. Magnus and R. Klar [13], A. Magnus and H. Roth [14]. The latter investigators were occupied with the study of an O_2-N_2 mixture on silica gel and revealed that the adsorption of O_2 in a mixture with N_2 proceeds more vigorously than in the

case of pure oxygen, while the adsorption of pure N_2 proceeds more successfully than from a mixture with O_2.

It may be noted that our present knowledge in the field of the adsorption of gaseous mixtures does not permit positive judgements in regard to the effect of one component on the adsorption of another component.

It seems probable that for a Kr-O_2 mixture we ought to expect a decrease of the adsorption value of oxygen, which is explained by the well-known law of displacement of the less adsorbable component (O_2) by the more adsorbable (Kr).

Conclusions

1. It has been shown possible to apply the adsorption method to the separation of krypton and xenon from a concentrate.

2. Measurements have been cited on the adsorption of O_2, Ar and Kr by silica gel for temperatures of -55, -93, and -120° and for pressures between 0 and 1000 mm Hg.

3. The basic factors of the process have been discussed with the purpose of showing its optimum parameters.

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[References to tables and their captions follow:]

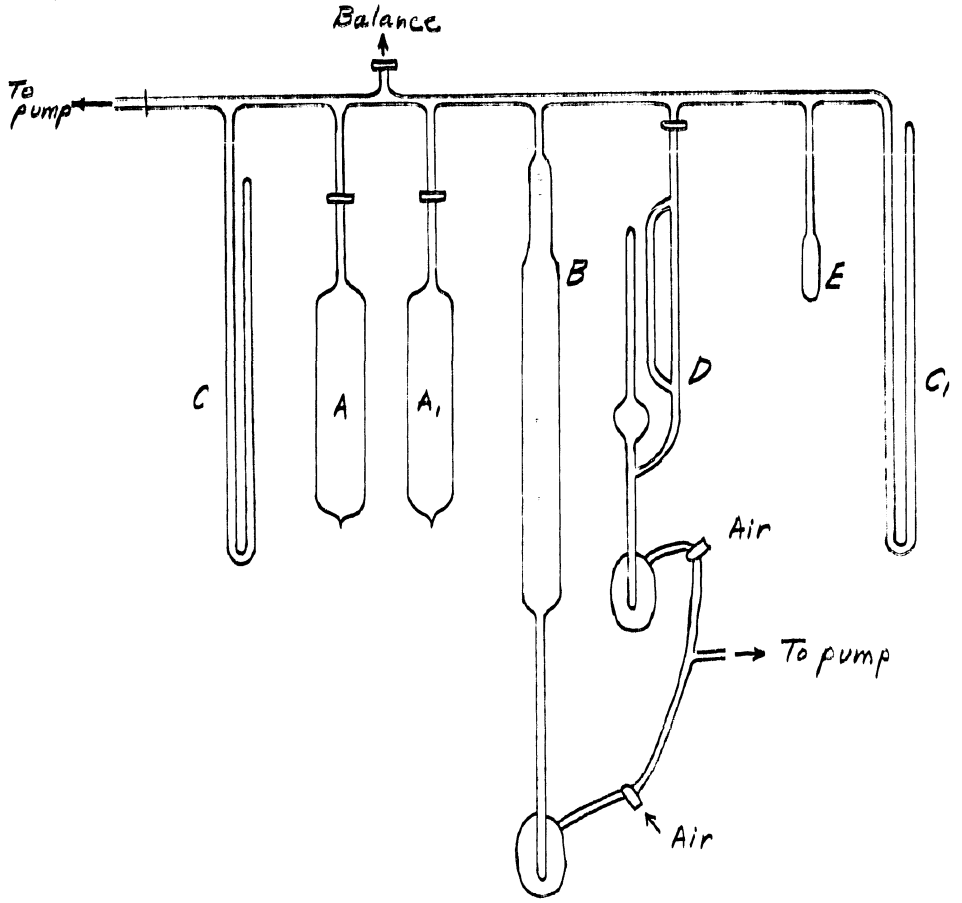

Figure 1. Diagram of the apparatus for studying the adsorption of gases.

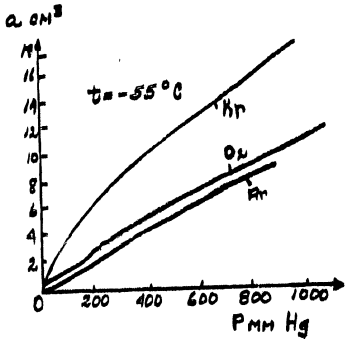

Fig 2

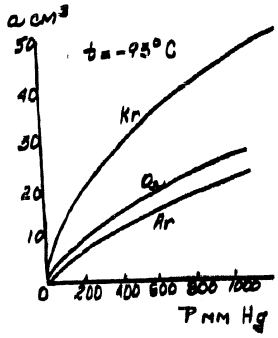

Fig 3

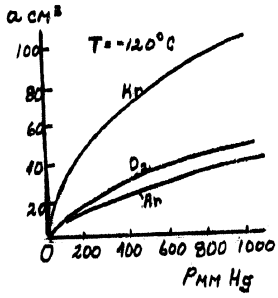

Fig 4

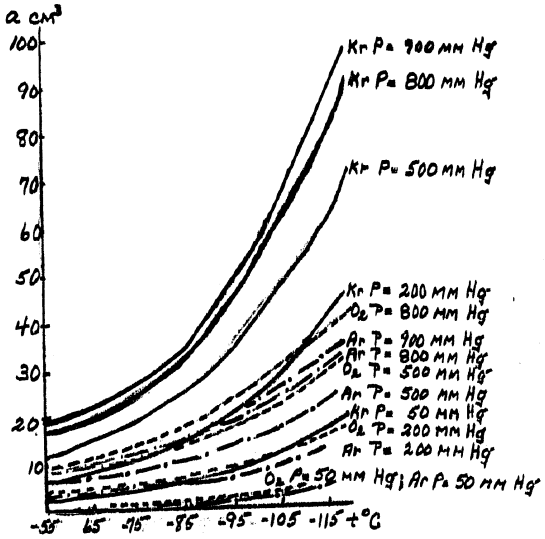

Fig 5

Table 1. Adsorption of Krypton

t° = -35°			t° = -55°			t° = -93°			t° = -120°		
Pmm Hg	a cm^3	a/p	Pmm Hg	a cm^3	a/p	Pmm Hg	a cm^3	a/p	Pmm Hg	a cm^3	a/p
15	0.856	0.057	19	0.654	0.034	1	0.27	0.27	4	5.434	1.35
60	0.907	0.015	33	1.25	0.037	9	2.31	0.26	16	10.229	0.64
77	1.398	0.018	69	2.462	0.035	26	4.91	0.19	33	16.26	0.49
110	2.099	0.019	120	3.652	0.030	55	8.00	0.14	53	22.845	0.43
151	2.974	0.019	190	5.49	0.030	98	11.52	0.11	108	32.625	0.32
212	3.933	0.018	280	7.54	0.027	153	15.50	0.10	213	46.925	0.22
276	5.163	0.018	398	9.70	0.025	229	19.97	0.08	400	64.725	0.16
367	6.553	0.018	521	11.74	0.022	324	25.07	0.08	649	82.875	0.12
483	8.283	0.017	666	13.85	0.022	464	30.84	0.06	912	98.526	0.11
608	9.423	0.015	802	13.53	0.019	712	38.28	0.05	1148	110.225	0.09
722	10.733	0.015	923	17.79	0.019	965	45.78	0.04	—	—	—
843	11.643	0.014	—	—	—	—	—	—	—	—	—

Table 2. Adsorption of Argon

$t° = -5\frac{1}{4}°$			$t° = -92°$			$t° = -120°$			$t° = -183°$		
Pmm Hg	a cm^3	a/p	Pmm Hg	a cm^3	a/p	Pmm Hg	a cm^3	a/p	Pmm Hg	a cm^3	a/p
5	0.1507	0.03	3	0.101	0.0339	3	1.059	0.353	1	0.08$\frac{1}{4}$	0.08$\frac{1}{4}$
7	0.1987	0.028	8	0.256	0.032	15	2.27	0.15	1.5	11.5$\frac{1}{4}$	7.7
10	0.1997	0.019	28	0.901	0.032	36	4.335	0.12	1.7	18.$\frac{1}{2}$9	10.8
78	1.05	0.013	67	2.132	0.032	73	6.$\frac{1}{4}$	0.09	1.9	30.87	16.3
1$\frac{1}{2}$5	1.66	0.011$\frac{1}{4}$	81	3.307	0.0$\frac{1}{2}$0	123	8.81	0.07	6.0	51.6	8.6
21$\frac{1}{4}$	2.56	0.0119	132	4.5$\frac{1}{4}$7	0.03$\frac{1}{4}$	168	12.0	0.07	2$\frac{1}{2}$	65.1	4.65
279	3.95	0.01$\frac{1}{4}$	191	6.232	0.032	300	1$\frac{1}{4}$.57	0.05	22	79.5	3.6
363	4.05	0.011	269	8.307	0.031	4$\frac{1}{2}$80	7.23	0.0$\frac{1}{2}$5	36	96.2	2.66
4$\frac{1}{2}$35	5.2	0.011	392	10.4$\frac{1}{2}$97	0.027	636	19.88	0.03	6$\frac{1}{4}$	119.0	1.86
527	5.31	0.0?0	4$\frac{1}{2}$98	12.60	0.025	802	22.66	0.028	107	1$\frac{1}{2}$0.8	1.32
627	6.4$\frac{1}{2}$9	0.010	610	1$\frac{1}{4}$.1$\frac{1}{4}$	0.023	910	28.3$\frac{1}{4}$	0.03	159	16$\frac{1}{4}$.5	1.03
736	8.08	0.0109	713	16.10	0.023	1039	30.29	0.028	211	187.2	0.89
960	9.87	0.010	779	18.4$\frac{1}{2}$6	0.02$\frac{1}{4}$				301	219.0	0.73
			868	20.05	0.023				38$\frac{1}{4}$	251.7	0.66
			1022	21.1	0.021				4$\frac{1}{2}$72	285.9	0.605
			11$\frac{1}{4}$8	23.03	0.020				539	323.3	0.6
									710	4$\frac{1}{2}$62.5	0.65
									800	635.8	0.79

Table 3. Adsorption of Oxygen

t° = -56°			t° = -92°			t° = -120°		
Pmm Hg	a cm^3	a/p	Pmm Hg	a cm^3	a/p	Pmm Hg	a cm^3	a/p
3	0.0723	0.024	4	0.3306	0.082	6	1.009	0.168
13	0.2504	0.02	17	1.0356	0.06	14	2.246	0.162
45	0.5814	0.013	44	2.52	0.06	30	3.979	0.132
106	1.4854	0.013	87	4.56	0.052	52	6.397	0.123
210	2.7964	0.013	150	7.18	0.05	125	13.202	0.106
318	4.3564	0.013	246	9.55	0.04	399	29.242	0.073
483	6.2769	0.013	365	12.88	0.035	529	37.19	0.07
678	8.17	0.012	506	17.55	0.034	781	41.51	0.053
866	9.90	0.011	738	21.44	0.027	973	45.55	0.046
1173	12.80	0.011	979	25.06	0.025	1148	49.86	0.043
			1122	28.39	0.025			
			1295	30.70	0.024			

Table 4. Adsorption Values for Different Volumes of Gas

a cm^3	t°	O_2 Pmm Hg	Ar Pmm Hg	Kr Pmm Hg	Kr -- Ar -- O_2
2	-55°	-150	195	60	1:3.2:2.5
	-93°	40	52	10	1:5.2:4.0
	-120°	10	10	--	--
4	-55°	300	360	130	1:2.7:2.3
	-93	80	120	20	1:6.0:4.0
	-120	30	25	--	--
6	-55°	475	555	210	1:2.6:2.3
	-93	130	185	35	1:5.3:3.7
	-120	50	45	10	1:4.5:5
9	-55°	765	875	360	1:2.4:2.1
	-93	215	305	65	1:4.6:3.3
	-120	90	90	15	1:6 :6.0
12	-55°	1086	875	545	1:1.6:1.6
	-93	310	305	100	1:3.05:3.1
	-120	120	90	23	1:3.9:5.2

Table 5. Effect of Pressure on Ratio of Components in Adsorbed Phase

Pmm Hg	Ar cm^3	O_2 cm^3	Kr cm^3	Ar -- Kr -- O_2
800	21	26.6	51.4	1:2.45:1.27
500	14.8	19.8.	40.8	1:2.77:1.34
200	7.9	10.2	23.6	1:2.98:1.29
50	2	3	10.2	1:5.1:1.5

- END -

Table 16. Effect of Temperature Fluctuations on the Mechanical Strength of Products Graphitized at 2200°.

No.	Kind of material.	Limiting stress = compression strength in kg/cm^2		
		a	b	c
2	50% of anthracite A + 50% of petroleum coke	146.0	112.0	77.0
7	Anthracite having a high ash content	136.0	102.0	-