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FLOTATION OF URANIUM ORES

A Thesis
Submitted to the Faculty of Graduate Studies
in Partial Fulfilment of the Requirements
for the Degree of
Master of Science
in Chemical Engineering
in the Department of Chemistry and Chemical Engineering
UNIVERSITY OF SASKATCHEWAN

by

Edward Brian Tinker

Saskatoon, Saskatchewan
April 1954

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ACKNOWLEDGMENTS

The author would like to express appreciation for the assistance and helpful criticisms offered by Dr. A. B. Van Cleave and Dr. S. D. Cavers throughout the course of this work.

Appreciation is also extended to the National Research Council of Canada for financial assistance in the form of a Bursary and to the Saskatchewan Research Council who provided funds for equipment and supplies.
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FLOTATION OF URANIUM ORES.

I. Introduction.

In the past few years the potential value of Northern Saskatchewan's low grade uraninite ores has increased greatly. In April of 1950 the value of acceptable uranium concentrates was raised from $2.75 per pound of $\text{U}_3\text{O}_8$ to a maximum of $6.00 per pound of $\text{U}_3\text{O}_8$ (1). With the present emphasis on the development of nuclear energy as a source of commercial power, the value of these ores may be expected to increase still further. In the present work an attempt has been made to apply froth flotation as a means of beneficiating one of these low grade ores.

From a consideration of the literature, it appears that at the present time, leaching is the most effective method of extracting uranium from its ores. However, leaching methods would probably become uneconomical if the grade of the ore were much below 0.10% $\text{U}_3\text{O}_8$.

Since the discovery of major deposits of uranium ores in Northern Saskatchewan attempts have been made at the University of Saskatchewan to beneficiate low grade uraninite ores by flotation (1, 2, 3, 4). These attempts met with little success except under conditions where the cost would probably be prohibitive.

Flotation is today the most popular method for beneficiating ores. The cost of flotation of sulfide ores based on 1940 prices ranges from 6.5 cents to 75 cents per ton of ore treated (5, p. 12, 101). The popularity of flotation is due in part to its low cost relative to many ore dressing procedures. However, in plants where tabling is used as a step prior to flotation, tabling costs seem to range from one-third to one-half of the flotation cost (5). While successful tabling is dependent on a large difference in the densities of the minerals fed,
In conducting the flotation research at the University of Saskatchewan, flotation has been regarded as a preliminary step to leaching. The emphasis has been on the achievement of high recoveries at the expense of concentrate grade. Much of the previous work at this university consisted of attempts to find a suitable collector for the uraninite in these ores. In the present work considerable attention has been directed towards the method of carrying out the flotation tests as well as to the search for a suitable collector. Several reagents have been found which seem to exhibit some degree of selectivity towards uraninite in the flotation process. In the present work the effect of variables such as pH, as well as the effect of the type of reagent used, have been investigated. As in the previous work, this investigation has been carried out with a view to obtaining high recoveries of uraninite in the concentrate. High concentrate grades have been of secondary interest.

II. Previous Research on the Beneficiation of Northern Saskatchewan Uranium Ores.

A. Flotation.

The majority of the flotation research on northern Saskatchewan uraninite ores has been conducted at the University of Saskatchewan. This research has been conducted mainly on ore from the Charlebois Lake region. Investigations of the flotation characteristics of other low grade uranium ores is being conducted by the Mines Branch in Ottawa and at Queen's University in Kingston. These researches are being carried out relatively independently.

Flotation studies on northern Saskatchewan uraninite ores were begun at this university by F. D. F. Talbot in 1951 (1). Talbot's
work, as well as that of I. J. O. Korchinski (2) in 1952, and G. A. Craig (4) in 1955, and the present author (5) in 1955, was conducted mainly with fifty gram Denver Sub-A flotation cells used as agitation cells without sub-aeration (see Section III, sub-section F.)

These cells have since been discarded as unsuitable for quantitative work. The results of Talbot's investigations showed that sodium oleate exhibited some selectivity towards uraninite in the flotation process. Using sodium oleate as collector, he also showed that the presence of Pb++, Fe++, Co++, Ni++, and Cr+++ has a definite effect on the recovery of uraninite. Talbot also concluded: (a) that potassium xanthate with sodium sulfide or Pb++ as activator is not effective as a collector for uraninite, (b) that sodium silicate and sodium sulfide depress uraninite in the presence of sodium oleate as collector, and (c) that there is some correlation between contact angles and the results of flotation tests. In addition, Talbot discovered that the ores do not affect the pH of distilled water and that the degree of secular equilibrium of the ore is not altered during the flotation process.

Talbot's work was continued by Korchinski in 1952 (2). Korchinski showed that whereas certain heavy metal cations, notably Fe+++ and Pb++, in small concentrations, act as activators for uraninite with sodium oleate as collector, excessive amounts of these ions depress the uraninite, under these conditions. The effect of particle size on the recovery and on the concentrate grade was also investigated by Korchinski. The finer fractions of the ore seemed to yield higher recoveries than were obtained on the unclassified material. Using sodium oleate as collector with lead acetate as activator Korchinski found that the flotation characteristics of the ore were not improved by either oxida-
tion with cold 50% hydrogen peroxide or by roasting at 860°C for 2.5 hours. Korchinski obtained recoveries approaching 50% using 1-dodecanethiol as collector. In this case the flotation medium was a 47.5% by volume solution of absolute ethanol in water.

Craig (4) and the present author (5) further investigated the use of thioalcohols as collectors for uraninite. Variation of the alcohol content of the flotation medium gave a maximum recovery at an ethanol concentration of 47.5% by volume. It was also noted that the character of the pine oil froth changed considerably with ethanol concentration. The froth became quite brittle at the higher alcohol concentrations.

The effect of mercaptan chain length and shape on the recovery of uraninite was also investigated. In this case the medium was water rather than the ethanol-water mixture. Maximum recovery was obtained with 1-hexanethiol as collector (5). Branching of the carbon chain seemed to have no effect on the recovery (4).

Craig attempted to concentrate the uraninite by removing the gangue by flotation. Typical cationic collectors, the Alamines and Alamac, supplied by General Mills Company, did not give encouraging results. Sodium metasilicate as a modifier with Alamine 26 as collector yielded a recovery of 49.4% at a grade of 0.16% U₃O₈ (4) from an ore analyzing 0.070% U₃O₈. Attempts were also made to depress the mica, since the uraninite is likely associated with it. These attempts met with little success. No improvement in the grade and recovery in the tailings was observed in attempts to activate the flotation of quartz and feldspar with Al⁺⁺⁺. Treatment of the ore with carbon disulfide and flotation with typical sulfide collectors gave a maximum recovery of 40% in the concentrate. Flotations carried out with Emcol X-25 as collector, with
and without attempted activation by lead acetate, were also conducted by Craig. The recovery was found to increase with the concentration of lead acetate, but when a recovery of 67.5% was obtained the percent $U_3O_8$ in the concentrate was the same as that in the original feed. Craig concluded that Emcol X-25 might be a suitable collector if suitable depressants could be found for other constituents of the ore.

In addition to flotation testing, other investigations of uranium ores have been conducted. These include radioautograph studies and determination of the grain size (1), behavior of the ores on grinding (2, 7) and contact angle measurements (1, 2). More complete details of these investigations may be found in References 1, 2 and 5.

Flotation research on uranium ores at Queen’s University has been relatively successful. These investigations were carried out mainly on euxenite ores with lead samples analyzing 0.10% $U_3O_8$. In several cases recoveries of 60%-80% of practically pure euxenite have been obtained (8).

Considerable research on the concentration of uranium ores by several methods is being conducted by the Mines Branch, Department of Mines and Technical Surveys, Ottawa (9). These workers concluded that Canadian uranium ores in general do not respond well to pretreatment by flotation. However, ores where the uranium is present as thuckolite or other hydrocarbon mineral likely would be beneficiated by flotation. The Mines Branch has also reported some results on the flotation of uranium ores containing carbonate (10). These ores generally show a high reagent consumption, possibly precluding an economically feasible flotation step (10).
B. Leaching.

Acid leaching of northern Saskatchewan uranium ores was briefly investigated by Korchinski (2). He concluded that while good recoveries could be obtained using hydrochloric acid and nitric acid in the leach solution, the process was probably too costly to be applicable to these lean ores. His work, and the work of B. Gunn, now in progress at the University of Saskatchewan, do indicate that leaching likely would be economical on material preconcentrated by flotation. Investigations by the Mines Branch have shown that recoveries as high as 97% can be obtained by leaching methods (9). Of the tests reported, only a few gave recoveries below 85%.

In general, two types of leaching processes have been investigated. Leaching by weak solutions of sulfuric acid is most common (11). This method is most suitable for silicious ores containing relatively small amounts of acid soluble gangue. Leaching by hot alkali carbonate solutions has been particularly popular for carnotite ores. This method has recently been applied to primary uranium ores such as pitchblende (12). The process is applicable to low grade pitchblende ores whose carbonate content prohibits acid leaching. The carbonate leaching process is somewhat more complicated than the acid leaching process, requiring higher temperatures and pressures. Many ores, including some of the ones found in the Beaverlodge area, respond equally well to either carbonate or acid leaching (11).

C. Gravity Concentration.

Korchinski (2) submitted a sample of a low grade uraninite ore to concentration by centrifugal classification and tabling. The material used in this single test did not respond well to gravity concentration.
The Mines Branch lists the following three types of ores as suitable for gravity concentration (9):

(a) Ores containing the majority of the uranium as unaltered uraninite or pitchblende and liberated by reduction to 10 mesh.

(b) Ores containing uraninite as well as columbates - tantalates of uranium.

(c) Complex pitchblende ores containing unaltered pitchblende as well as heavy arsenite and sulfide minerals.

Jigging, a gravity concentration method, is used in the treatment given the pitchblende deposits at Port Radium. The majority of the research on the gravity concentration of Canadian uranium ores is being conducted by the Mines Branch and the University of Alberta. Results of the Alberta experiments are not available. Recoveries as high as 90% have been obtained in a few cases by the Mines Branch (11).

III. Flotation Theory and Practice.

A. Definition of Terms.

"Flotation includes any operation in which one solid is separated from another by floating one of them at or on the surface of a fluid" (6). It is possible, by flotation, to separate materials regardless of their densities since separation by flotation is based on surface properties. In its simplest terms, flotation involves the separation of one or more solid constituents from a pulp of the solid material and a liquid phase by causing the desired material to adhere to air bubbles. These bubble-particle aggregates rise through the pulp and are swept off at the top of the flotation cell. The basis of the process is the stability of the solid particle at the gas-liquid interface. The fundamental criterion for the stability of a solid particle at such an
interface is the existence of a finite contact angle (13)

The contact angle is best defined graphically, as in Fig. 1, where \( \theta \), the contact angle, is measured through the liquid phase. In practice, different contact angles are observed, depending on which phase is being displaced from the solid surface. The receding contact angle is measured when the gaseous phase is displacing the liquid phase. When the gaseous phase is being displaced by the liquid phase the advancing contact angle is measured. For stable flotation it is the receding contact angle which must be finite (13).

Natural mineral surfaces are usually wet by water; that is, the contact angle is zero. Paraffin, on the other hand, gives the largest contact angle yet measured, 105° (15). The purpose of the collector in flotation is to establish a hydrocarbon coating on the desired mineral. Such a hydrocarbon coating will generally exhibit a finite contact angle.

The next step in the flotation process is the attachment of the hydrocarbon-coated mineral particle to a rising gas bubble. Obviously, the capacity of a flotation process will depend on the area of gas-liquid interface present. Air is introduced into flotation pulps and dispersed in the form of fine bubbles. The frother is present to prevent coalescence of these air bubbles.

In practice, other reagents known as modifiers, are useful in improving the selectivity and efficiency of flotations. These modifying agents function by reaction with or adsorption on the surface of the solids present, thereby altering their surface characteristics.

Generally speaking the flotation process divides an ore into two fractions. The fraction of the ore containing the desired constituents is termed the concentrate, whereas the portion containing the worthless material is referred to as the tailings (14). It is usual
SURFACE FORCES IN FLOTATION

Fig. 1.

Gas

Mineral

Liquid

$\gamma_{sl}$

$\gamma_{sg}$
for the concentrate to be removed in the froth and for the tailings to be left in the cell at the end of the process. The valuable material is referred to as the minerals while the undesirable material is called gangue (14).

Often the fine portions of the ore are removed by settling of the coarser fraction and decantation of the fluid containing the fines. The reasons for this will become apparent in later sections of this work. These fine portions of the ore so removed are generally termed slimes.

Flotation operations may be broadly divided into roughing, cleaning, and scavenging steps. Each step in itself represents a complete flotation. As the name implies, the roughing flotation constitutes a rough separation of the ore into concentrate and tailings. The concentrate from this operation is cleaned and the tailings are scavenged. Schematic flow sheets and more complete discussions of the operations may be found in References 5, 6, or 14.

In order to concentrate an ore by flotation, it is first necessary to liberate the minerals from the gangue. It is desirable that each particle should be composed of either minerals or gangue. Such a situation is termed 100% liberation.

The effectiveness of a flotation operation is usually described by the grade of the concentrate and the percent recovery of the valuable constituent in the concentrate. For the purposes of this work grade will be defined as the percent by weight of the valuable constituent and recovery as the percent of the total valuable constituent recovered in any one fraction.

B. Theories of Collector Action.

The function of the collecting agent has been described in the preceding section. Many theories have been advanced to explain the

See p. 22
selective coating of mineral surfaces by flotation reagents (5, p.12-14)

The earliest attempts at flotation involved the use of oily or fatty reagents (14). Taggart (5, p.12-06) describes coating by oily collectors as follows:

"When coating is affected with undissolved neutral oily liquids, the action occurring between the solid surface and the coating agent is one of mutual solution, and the coating formed is a solution of the two substances."

The validity of this postulate is evidenced by the fact that these neutral oily liquids are generally not collectors for substances which they do not dissolve (5, p.12-06).

In the early 1920's discoveries by Perkins (15) and Keller in a patent to Minerals Separation Company (14) showed that oils were not essential for flotation. Their discoveries mark the beginning of what has been called chemical flotation, to distinguish it from the earlier oil flotation (14). The first formulation of the chemical theory of flotation was given by Taggart, Taylor and Ince (16) as follows:

"All dissolved reagents which, in flotation pulps, either by action on the to-be-floated or the not-to-be-floated particles affect their floatability, function by reason of chemical reactions of well recognized types between the reagent and the particle affected."

It is important to realize that this theory and that of mutual solution are complementary rather than contradictory for they apply to different types of reagents.

Reagents which function by chemical reaction with the surface are nearly all organic acids, bases, or salts. To function as collectors these compounds must be slightly soluble in water and, in the case of acids and salts at least, ionizable in an aqueous medium (5, p.12-06).
The basic type of collector (amines and amine salts) need not be ionized. The part of the molecule combining with the surface must contain the hydrocarbon radical and the compound formed between the collector and the surface must be insoluble under conditions existing in the pulp zone adjacent to the mineral surface (5, p. 12-05).

Considerable experimental evidence is available in confirmation of the chemical theory of collection. Perhaps the best of this evidence is the fact that ion exchange occurs when minerals are leached with collector solutions. Taggart (5, p. 12-05) found that when such leaching is carried out there is a decrease in the concentration of collector ions and a corresponding increase in the concentration of mineral surface ions of the same charge. For the reaction between xanthate solutions and slightly oxidized galena surfaces Taggart, Taylor and Knoll (17) have shown that the removal of xanthate from the solution is accompanied by an increase in the concentration of sulfate and hydroxyl ions in the solution. The reactions probably proceed as follows (13):

\[
Pb(OH)_2 + 2K-S-C-O-C_2H_5 \rightarrow Pb(S-C-O-C_2H_5)_2 + 2KOH
\]

\[
PbSO_4 + 2K-S-C-O-C_2H_5 \rightarrow Pb(S-C-O-C_2H_5)_2 + K_2SO_4
\]

If strict precautions are taken to prevent oxidation of the surface there does not appear to be any reaction between the xanthate and the lead surface (18). This means that some surface oxidation is necessary in the flotation of galena by xanthate. Excessive oxidation of the surface is unfavorable to the flotation of sulfide ores (15). The formation of lead ethyl xanthate on the surface of galena likely results in the orientation of the collector film as follows: (5, p. 12-05):
The mechanism of the xanthate flotation of sulfide minerals seems fairly well understood. The reaction of other collectors with other types of mineral surfaces has not been so thoroughly studied. It is possible that physical adsorption contributes to the coating action of the collector (6, 14).

Chemical collectors are usually classified as anionic or cationic. In the anionic type the hydrocarbon radical is in the anion. Organic acids and salts comprise this class. Common examples are the xanthates and thiophosphates:

\[ \text{E-O-S-M} \]

A xanthate

\[ \text{R-O-PS-M} \]

A thiophosphate

where R represents a hydrocarbon radical and M is usually an alkali metal ion or the ammonium radical. Other reagents which fall into this category are thioalcohols, thiocarbanilid, diphenylthiocarbazid, mercaptobenzothiazol, organic sulfides, carboxylic acids and their salts, and sulfox compounds.

The cationic type of collector is much less common than the anionic type. In this type of collector the hydrocarbon radical appears in the cation. Examples are the amines and -onium compounds (5, p. 12-04).
These -onium compounds generally contain pentavalent nitrogen or phosphorus or tetravalent sulfur. In the amine class, laurylamine and α-naphthylamine have been used as collectors. Several of the Emulsol® collectors are cationic reagents of the -onium type.

C. Frothers and Frothing.

The function of the frothing agent in flotation pulps is to impart stability to the froth formed by the introduction of gas. Frothing agents concentrate at the interface between gas and liquid according to the Gibbs equation:

\[ a = - \frac{\partial \gamma}{RT \partial \alpha} \]

where \( a \) is the excess concentration in the surface layer, \( \gamma \) is the surface tension, \( \alpha \) is the activity of the solute, \( R \) is the gas constant, and \( T \) is the absolute temperature. This equation predicts that substances which lower the surface tension tend to concentrate in the surface layer. Such substances are termed surface active.

There is no theory accounting for all the factors which influence the stability of froths (19). A theory due to Foulk (21, 21, 22) emphasizes that the spontaneously produced concentration difference between the surface and the bulk of the fluid requires the expenditure of work in its elimination.

The presence of partially wet solid particles at the gas-liquid interface is an aid to froth stability (19). The effect is analogous to fluid viscosity (5, p. 12-41). Deformation of bubble walls is accompanied by interaction between mineral particles adhering to the bubble and other particles in suspension in the bubble walls. Such interaction is essentially a resistance to deformation, hence an aid to stabilizing

* Products of the Emulsol Corporation, 59 E. Madison St., Chicago, Illinois.
froth.

In the absence of solid particles in the gas-liquid interface two factors are probably operative in the stabilization of froths. A high surface viscosity may cause the formation of a stable froth (24). The decreasing stability of froths with increasing temperature is likely due to this factor (5, p.12-42). In cases where a high surface viscosity does not exist, the stability is probably due to the difference in surface tension between the surface fluid and the bulk of the fluid (24).

The effect of electrolytes on the froth varies with the type of electrolyte. Neutral salts generally have no effect unless they destroy the frothing agent by chemical reaction. Alkalis usually increase frothing. Excess alkali generally results in underloaded, gangue bearing froths. Acids tend to decrease froth volume likely because of the flocculating action on slime gangue (5, p. 12-42).

It would seem that heavily mineralized small bubbles would offer the maximum capacity in a flotation process. Many small bubbles offer a greater surface area than do a few large bubbles. In practice it is important that the bubbles be not too small. Over-mineralization is also detrimental to the process. The bulk density of small, heavily mineralized bubbles may become greater than the bulk density of the pulp. In this case the bubble-particle aggregate will not rise to the surface where it may be swept off. Heavily mineralized bubbles also tend to trap gangue particles by a screening action resulting in reduction in the concentrate grade. It is advantageous to have a froth in which coalescence is occurring in the upper layer (5, p. 12-42). This permits the release of entrained gangue particles. Ideally the froth should be stable only as long as it remains in the flotation cell.

In order that collection and frothing be controlled indepen-
dently, the ideal frother should have no collecting power. It should be highly surface active so as to produce frothing in low concentration and should function independently of other solutes present in the pulp (5, p. 12-45). Good control of frothing is essential in carrying out separations of similar substances. This is accomplished by careful choice of the type of flotation machine and careful control of the rate of feed of reagent and ore (5, p. 12-46)

D. Levitation - The Contact Angle.

"The attachment of air bubbles to collector coated mineral particles and the subsequent separation of the air-mineral aggregates from the non-bubble bearing particles by differential sedimentation and skimming of the float is called levitation." (5, p. 12-57)

The main theories attempting to explain the mechanism of bubble particle attachment are the gas precipitation hypothesis of Taggart (5, p. 12-39) and the direct contact hypothesis (14). Although these theories apply specifically to the agitation type of flotation machine where collection occurs essentially in the main body of the pulp, the direct contact hypothesis also accounts for the mechanism of collection in pneumatic and sub-aeration machines (14). In pneumatic machines collection occurs mainly in the bubble column, whereas in the sub-aeration type bubble-particle aggregates are formed both in the bubble column and in the body of the pulp (14).

The gas precipitation hypothesis of Taggart proposes that the gas is selectively expelled from solution at non-polar surfaces (5, p. 12-39). In the agitation type of machine the pulp is kept continually saturated with gas (5, p. 12-75). Pressure is built up ahead of the impeller causing solution of the gas in the liquid. Behind the impeller a vacuum is formed, varying from five to eight inches of mercury (14). Gas which was dissolved at the increased pressure ahead of
the impeller will come out of solution at the reduced pressure behind the impeller.

The direct contact hypothesis appears to offer more comprehensive treatment of the mechanism of bubble-particle attachment. This treatment explains collection in the three types of machines previously mentioned, whereas the gas precipitation hypothesis seems to account for the experimental results only in agitation machines (14). The reduced floatability of very fine particles is accounted for by the direct contact hypothesis. The gas precipitation hypothesis offers no explanation for this phenomenon (14). Gaudin has shown, for a very limiting case, that in order for a rising bubble to encounter a falling mineral particle the bubble and the particle must be essentially one above the other (14). That this is for a very synthetic case is evidenced by the assumptions made in the derivation:

"(a) The water is assumed to be incompressible and to have no viscosity.
(b) The water is assumed to be infinitely divisible.
(c) Bubble and particle are assumed to be spherical.
(d) Bubble and particle are assumed to be the only disturbing factors in an otherwise stationary system.
(e) Bubble and particle are assumed to have an irrotational motion."(14)

Gaudin has applied these assumptions to an estimation of the relationship between relative size of bubble and particle and probability of encounter (14). He concluded that in situations where the particle is relatively small compared to the size of the bubble, the probability of encounter varies directly as the size of the particle. This predicts the reduced floatability of very fine particles.

While the direct contact hypothesis appears to offer the best
explanation for the mechanism of bubble-particle attachment, Taggart (5, p. 12-59) doubts that sufficient force and time of contact exist in flotation pulps to effect attachment of mineral particles to pre-formed bubbles except on rare occasions. On the other hand, Gaudin (14) expresses doubt that heavily mineralized bubbles could be formed by gas precipitation.

Russian workers have recently derived expressions for the maximum time of contact of bubble and particle and for the weight of the mineral load carried by the bubble (25). The maximum time of contact, \( \tau \), is given by:

\[
\tau = 1.8 \frac{R}{u}
\]

where \( R \) is the radius of the bubble and \( u \) is the relative velocity of bubble and particle. The time, \( \tau \), is calculated to be approximately 0.004 - 0.008 seconds. The time during which adhesion may occur in a flotation cell is the time required for the particle to pass from the top to the equator of the bubble (25). The weight of mineral particles, \( q_v \), adhering to a bubble is given by

\[
q_v = c \alpha_1 h \left( 1 + \frac{U_p}{U_l} \right) \left[ R \cos \left( \frac{U_l t_i}{R} \right) + l_1 \right]^2
\]

where \( c \) is the concentration of particles in the pulp, \( \alpha_1 \) is the partial content in the solid phase of particles having an adhesion time of \( t_i \) (taken as the time of contact required for adhesion to occur) and a radius of \( l_1 \), \( h \) is the height of the flotation cell and \( U_p \) and \( U_l \) are the velocities of the bubble and of the particle, respectively. The selectivity of the flotation process depends on the relationship between \( t_i \) and \( \tau \) (25).

The selectivity exhibited in flotation processes is explained by Taggart on the basis of selective precipitation of gas at non-polar
surfaces (5, p. 12-59). Gaudin, who favors the direct contact hypothesis, explains selection by the difference in wettablility of polar and non-polar surfaces (14). Direct encounter between a bubble and a polar particle is hindered by the presence of hydrated ions at the surface of the particle. If such encounter does occur the liquid will tend to displace the gas on the surface and the bubble will not adhere. If a bubble encounters a non-polar particle, the gas will tend to displace the liquid and the bubble will adhere.

For a time it was thought that electrostatic force played a role in the selection occurring in flotation processes (26, 27, 28). The attachment of bubbles and floatable particles was believed due to electrostatic charges of opposite sign present on the bubble and the particle. According to Gaudin, the presence of electrostatic charges in the conducting solutions encountered in flotation is impossible (14). McBain and Williams have shown that only a small number of free electrostatic charges are present at the surfaces of gas bubbles and oil droplets in water (29). The theory of electrostatic attraction as a factor in the selectivity in flotation has largely been discredited (14).

The relationship among the forces involved in the static bubble-particle equilibrium is given by the Young equation:

$$\gamma_{LS} + \gamma_{LG} \cos \theta = \gamma_{GS}$$

where $\gamma_{LS}$, $\gamma_{LG}$ and $\gamma_{GS}$ are the interfacial energies of the liquid-solid, liquid-gas, and gas-solid interfaces, respectively. Fig. 1 shows the relationship of these forces. The work done in removing a bubble from a unit area of solid surface is given by

$$W = \gamma_{LG} + \gamma_{LS} - \gamma_{GS}$$

since, in removing the bubble the destruction of the solid-gas inter-
face is accompanied by the formation of liquid-gas and liquid-solid interfaces. In this equation \( \theta \) represents the interfacial energies per unit area. Introducing the Young equation into the expression for the work required to remove a bubble yields

\[
W = \gamma_{LG}(1 - \cos \theta)
\]

An increase in \( \theta \) results in an increase in \( W \). It is for this reason that large contact angles are desired in flotation. This equation also predicts that when \( \theta = 0 \), \( W = 0 \), and the bubble should not adhere.

The final act in levitation is the rise of the bubble-particle aggregate through the pulp and its removal as froth. In order for the aggregate to rise, its bulk density must be less than that of the flotation pulp. Thus pulp density becomes an important variable in flotation. Gaudin (14) has derived an equation for the lifting power of a bubble in terms of the gas-liquid interfacial energy and the size of a single particle

\[
\eta_{LG} = \frac{V(D - d)}{L}
\]

where \( V \) is the volume of the particle of density \( D \), \( d \) is the bulk density of the pulp and \( L \) is the length of the wetted perimeter of the solid. Wark tested the validity of this equation by loading a hydrocarbon coated zinc plate. He found the lifting power to be several times that predicted by the equation,(5, p. 12-57).

As will be seen from the preceding discussion the mechanism by which bubble and particles become attached is a matter of some conjecture. Considerable evidence has been cited in support of both the direct contact and the gas precipitation hypotheses (5, p. 12-37 to 12-41; 14).
E. Modifying Reagents.

Often flotation results are greatly improved by the use of certain modifying agents in conjunction with the collectors and frothers. These reagents may function as pH regulators, activators, or depressors. Among the common reagents used to control the pH of the pulp are calcium hydroxide, sodium hydroxide, sodium carbonate, sulfuric acid, and hydrochloric acid.

Activators function, as the name implies, to enhance the floatability of certain constituents of the ore. The activating influence is generally exerted by chemical reaction. It usually involves the formation of a compound less soluble than the substances constituting the mineral surface. Thus, activating ions for zinc sulfide are those which form less soluble sulfides (15). Compounds formed between the collector and the activating agent must be less soluble than compounds formed by a corresponding reaction between the collector and the mineral surface (14). The common metals whose organic salts and oxides are the least soluble are copper, lead and mercury. Salts of these metals are the most effective activators (14).

Depressors function in opposition to activators to suppress the flotation of one or more undesirable constituents. They form at the mineral surface a compound less amenable to flotation than the constituents of the mineral surface. According to Taggart (5, 12-25) this function may be accomplished in various ways. Ions likely to cause accidental activation may be precipitated or complexed by the depressor. Accidental activation is uncontrolled activation caused by the presence of soluble salts in the pulp. Depressors may function by complexing the surface ions, thus reducing their concentration and inhibiting their reaction with the collector. Some depressors exert
their influence by destroying the collector coating. In some cases depression may occur through the formation of a hydrophyllic coating at the mineral surface.

Frequently such reagents as sulfuric acid or sodium hydroxide are used as cleansing agents in a scrubbing step ahead of the flotation. This is done to insure that each constituent presents its normal surface to the flotation reagents. Sulfide ores were at one time floated in acid media to cleanse their surface of oxide coatings (14).

Slimes frequently interfere with flotation operations. This interference may occur in one or more of the following ways:
(a) Increasing the consumption of reagents,
(b) Production of an uncontrollable amount of froth, frequently referred to as overfrothing,
(c) Contamination of the concentrate in the case of slime gangue, and
(d) Reduction in the recovery.

The latter two are the direct result of coating of the mineral particle by slime gangue. De Guidice (30) postulated that, in slime coating of galena by calcite, lead carbonate is formed which acts as a binding cement between the calcite particles and the galena. According to Banhoff slime coating occurs through flocculation (31).

Dispersing agents such as alkali silicates and phosphates, inorganic acids, alkalis, and organic dispersants are frequently used to prevent slime coating (5, p. 12-22). Slime coatings may be removed by violent agitation of the pulp. Known as attrition mixing, this treatment utilizes the abrasive action of the particles on one another to remove slime coating. Slimes may also be removed by allowing the "sand" to settle and decanting the fine suspended portion.
E. Types of Flotation Cells.

Gaudin classifies flotation cells on the basis of the method of introduction of gas (14). This classification divides flotation cells into: (a) agitation cells, (b) sub-aeration cells, (c) cascade cells, and (d) pneumatic cells.

In the agitation type of flotation cell, gas is introduced into the pulp through the vortex formed by the rapidly rotating impeller. The sub-aeration type differs from the agitation type in that the gas, drawn in by the impeller, is delivered directly to the impeller and dispersed into the pulp (5, p. 12-63). This is often accomplished by surrounding the impeller shaft with a hollow pipe through which the gas is drawn (as in the Denver Sub-A cell). Cascade cells accomplish the introduction of gas through the tumbling action of the pulp. This is generally accomplished by allowing a stream of water or pulp to plunge into a body of pulp (5, p. 12-61). The gas supply to pneumatic cells is delivered under pressure. The gas is dispersed as bubbles by forcing it through a porous medium in the bottom of the flotation cell.

In addition, combination machines have been designed which employ more than one mode of gas introduction.

In pneumatic cells the pulp must be fully conditioned with reagents prior to flotation (6). Due to the relatively mild agitation in pneumatic cells they produce a cleaner concentrate (6). Mechanical cells, in which there is direct mechanical agitation, are more efficient in reducing the grade of the tailings although they yield a concentrate more contaminated with gangue (6). More complete details of the methods of operation of these types of cells, and descriptions of particular machings are provided by Taggart (5, p.12-55 to p.12-78),
Gaudin (14), Brown (6), and Simons (32).

G. Flotation Testing and the Correlation of Batch Tests with Commercial Operation.

Flotation testing may be conducted to investigate the characteristics of an ore, to determine the effects of a particular reagent on a variety of ores, to investigate the operating characteristics of certain equipment, or to investigate a flotation circuit or process (5, p. 19-174) (14). In conducting flotation tests, it is imperative to duplicate as closely as possible conditions existing in commercial plants. The first step in the test procedure should be a chemical and microscopic analysis on a representative sample of the ore (14). This will yield data on the composition, structure, and associations of the minerals in the ore.

Grinding procedure should be as close as possible to that used in commercial plants. The general procedure in testing is to dry grind to -10 mesh and wet grind in a ball or rod mill to flotation size. Batch grinding is usual (5, p. 19-179). Dry grinding is generally unsatisfactory. In one case a dry ground ore yielded a recovery of only 60% in the flotation process. The same ore when wet ground gave a recovery of over 90% (33). The major difference between the usual laboratory crushing and grinding procedure and the size reduction process used in commercial plants is in the size of the feed to the ball mill. In commercial plants this is rarely less than one quarter inch (5, p. 19-179).

The usual procedure followed in flotation testing is given by Taggart (5, p. 19-180) as follows: The product of the ball mill is transferred to the operating flotation cell with the air valve closed. Conditioning agents other than activators and depressors are now added and the pulp conditioned for a short length of time. The addition of
other conditioning agents is again followed by a short period of agitation. Next, the collector and frother are added in that order and another short agitation period allowed. Water is then added to bring the pulp to the proper dilution, the air valve is opened, and collection carried out either for a specific length of time or until an end point, as determined by the character of the froth, is reached. The froth is normally skimmed by a slowly rotating paddle. In roughing operations the froth is skimmed deeply whereas in cleaning operations shallower skimming is practiced. Occasionally provision is made for the continuous addition of water containing some frother in order to maintain the pulp at a constant level. A low grade concentrate and a clean tailing may be obtained by rapid and deep skimming. Slow, shallow skimming will produce a finished concentrate and a high grade tailing. By practicing first shallow and then deep skimming the sample may be separated into a finished concentrate, a middling fraction, and a low grade tailing.

Workers at the U. S. Bureau of Mines devised a locked test to reduce the middling fraction (54). The flow sheet for such a test is shown in Fig. 2. In this test two stages of cleaning are used and the middling fraction is not normally reground. However, locked middling may be reground and returned to the flotation cells. This procedure frequently interferes with reagent control (5, p. 19-181). The products of the various stages of the test are shown in the flow sheet. The middling fractions (tailings from 4C, 4RC, and 4S) are routed to the tailings. These may be recovered as middlings as indicated by the dotted line leading to the product M. The tailings from 2S and 3S also contain middlings. This test bears no resemblance to any mill flow sheet. Taggart doubts whether this procedure approximates mill conditions any more closely than the standard batch test (5, p. 19-181).
FLOWSHEET FOR LOCKED TEST:

New Feed Batcher Flotation Operations.

Products

R: rougher period
S: scavenger period
C: cleaner test
RC: re-cleaner test

Fig. 2 (after Taggart)
Recently a method has been devised for reducing the number of flotation tests required for the investigation of a given set of variables. It is known as the factorial design method. "Factorial design is a mathematical method of drawing valid conclusions from a series of tests made in a predetermined pattern" (35). To test a number of variables by the usual statistical methods would require a large number of experiments. By making use of one result several times the method of factorial design reduces the number of tests to a minimum (35). If a variable is tested, say at two levels, the method affords a means of statistically evaluating the significance of the difference between the two results. The calculations will also indicate the possibility of interaction between the variables. The basic requirement for the application of the factorial design method to the planning and interpretation of an experiment is that the experimental results follow a normal distribution (35). The scheme followed is to test each variable at two levels. If \( n \) variables are to be investigated, \( 2^n \) tests are required to cover all combinations of the variables (36).

The final step in flotation testing is the correlation of batch tests with commercial operation and the prediction of results to be expected on a plant scale. The translation of laboratory results to plant operation is easier in flotation than in most other chemical and metallurgical operations (5, p. 19-184, 14). Primarily this ease of translation is due to the fact that the number of particles involved in a charge as small as 500 grams is large enough that statistical averages hold reasonably well (14). Flotation feed will generally be coarser in the plant than in the laboratory (5, p. 19-184). Testing is usually conducted in distilled water, and the presence of
soluble salts present in plant water may affect the performance (5, p. 19-185). Some batch tests using undistilled water are probably advisable in order to determine the effect of dissolved salts. If recirculation of water is practiced in the plant, reagent consumption will likely be lower than indicated by batch testing (5, p. 19-185). If the plant operation follows the usual flotation flow sheet (see References 5, 14, 3 or 33), the concentrate grade and recovery will approximate that obtained in a cleaning flotation in the laboratory, whereas the concentration of the tailings will be similar to that obtained in the laboratory roughing operation (5, p. 19-185).

Good plant design requires an accurate knowledge of circulating loads which is difficult to obtain from batch tests (14). Inspection of a typical flotation flow sheet will show that almost all flotation operations involve the production of intermediate products which are returned to the circuit. If the intermediate product is identical in particle size, composition and state of liberation with the new feed at the point of introduction, circulating loads can be calculated from batch tests (14). New feed is taken to mean material, other than that being recycled, being added to the flotation circuit at a particular point. If the single pass mass fraction of the intermediate product is a fraction, $1/p$, of the new feed at the point of introduction, the steady state mass fraction of the circulating load will be the fraction $1/(p-1)$, of the new feed at that point (14). In general, the intermediate product is not identical with the new feed, and the circulating load is actually higher than predicted by these considerations (14).

The gap between batch tests and commercial operation may be bridged by the continuous type of laboratory testing followed by
pilot plant testing. The major uncertainty in the conversion of pilot plant data to plant operations is in the accurate prediction of costs (14).

IV. EXPERIMENTAL

A. Description and Preparation of Ores Tested.

The uranium ores used in the flotation tests reported in this work were obtained from the Charlebois Lake area in northern Saskatchewan. The general geology of the area has been discussed by Mawdsley (57). The most common occurrence of radioactive minerals in the area is in a fine pegmatite. This rock is typically light grey to white when composed mainly of feldspar and some quartz. A darker variety composed principally of mica and smoky quartz is also noted. In some of the main radioactive zones a pinkish type of pegmatite occurs. The grain size of this fine pegmatite varies from 0.04 inches up to 2 inches (57). From analyses carried out on seventeen samples, Mawdsley concluded that a rough relationship between the quartz content and the radioactivity existed (57). Although the uraninite is generally found in close association with the biotite, there is no distinct relationship between the biotite content and the radioactivity (57). As found by Mawdsley the major constituents of the rock are quartz, oligoclase, orthoclase and biotite with smaller amounts of accessory minerals often present.

The flotation tests in the 50 gram Denver Sub-A cells were conducted with an ore sample numbered DE52-2. This sample is from the same ore as DE52-1, but was ground at a different time. Sample DE52-1 was used by Craig (4) and the present author (5) in previous research on this project. These samples were crushed in a Braun "Chipmunk" laboratory jaw crusher and dry ground in a Cave vertical plate mill. The fraction of the ground ore passing a 10 mesh Tyler standard screen
was used in the flotation tests. The screen analyses of the -10 mesh portions of these samples are given in Table I and Figure 3.

Table I. Comparative Screen Analyses of Ore Samples DE52-2 and DE52-1.

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<tr>
<th>Tyler Standard Screen Mesh</th>
<th>Aperture, inches</th>
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<th>DE52-2</th>
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A representative sample of ore DE52-2 analyzed 0.054% $U_3O_8$ by the tray method (Section IV, Sub-section 6). Craig, using the thyrode tube method found the ore assayed 0.0567% $U_3O_8$ (38).

Ore sample DE52-1 was used in the majority of the flotation work reported by Craig (4). Sample DE52-2 was used in a few tests. The +10 mesh fractions of both samples were rejected on the basis that they contained very little activity. However, this conclusion was based on sample DE52-1, which was more finely ground than sample DE52-2. In

* These analyses were done on the -10 mesh portion of the ore.
the present work a thorough investigation of the activity of the +10 mesh portion of sample DE52-2 was conducted. The mica particles were hand picked from a sample of +10 mesh portion of this ore. The mica fraction and the residue, consisting mainly of quartz and feldspar, were ground separately in a mortar. The material passing a 40 mesh screen was analysed by the tray method. The mica fraction analysed 0.25% $\text{U}_3\text{O}_8$, whereas the feldspar-quartz fraction analyzed 0.05% $\text{U}_3\text{O}_8$. This result bears out previous conclusions that the uraninite is associated with the mica in these ores (37). The rejection of the +10 mesh portion of sample DE52-1 by Craig on the basis of its low activity seems in order. In the flotations of sample DE52-2 reported in this work, the +10 mesh fraction was still rejected since it probably consisted of particles too large to float.

The flotations in the 500 gram Denver Sub-A cell were carried out on an ore sample designated DE55-1. This was Charlebois Lake ore obtained from the Mines Branch, Department of Mines and Technical Surveys, Ottawa. Approximately 700 pounds of this ore were crushed in the crushing rolls located in the Ceramics Laboratory at the University of Saskatchewan. The rock was fed through the rolls twice. On the final pass the rolls were spaced approximately 0.05 inches apart. A screen analysis of this crushed sample was conducted by Thompson (39). The results are reported here in Table II and Figure 4. It was intended that this coarse material be prepared for flotation by wet ball milling immediately prior to flotation. Several tests were conducted in order to determine the length of grinding time necessary to produce, from this crushed material, a feed suitable for the flotation cell. In all tests the charge to the ball mill consisted of 700 grams of ore, 350 ml. of water, 5 pounds of 0.75 in. nominal diameter steel balls, 5 pounds of
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1 in. nominal diameter steel balls and 5 pounds of 1.5 in. nominal diameter steel balls. The ball mill was rotated at 54 revolutions per minute for the desired time. The following grinding tests were conducted: one in which the grinding time was 120 minutes, one in which the grinding time was 45 minutes and three in which the grinding time was 20 minutes.

* For the effects of wear on the balls see Appendix A.
time was 20 minutes.

The screen analysis for the 120 minute grind is not available as extreme difficulty was encountered in attempting to carry out the screen analysis of this material. Even after prolonged drying, the material tended to agglomerate on the screens. Workers at the Mines Branch suggested a combination wet and dry screening procedure to overcome such difficulties (40). This procedure involves washing the entire sample over the finest screen in the series. The fine material is flushed through the screen. The coarser material may be recovered from the screen, dried, and screen analyzed in the normal way. In the procedure received from the Mines Branch a 525 mesh Tyler standard screen was the finest used. In the screen analysis reported in this work a 400 mesh Tyler standard screen was the smallest screen size used. It was found that even distilled water passed through the 400 mesh screen with difficulty. The screen analyses of the other four tests are reported in Table II. The 45 minute grind and the first 20 minute grind are shown graphically in Figure 4. Assuming that liberation was fairly complete at 65 mesh (1), it was decided that 20 minutes ball milling under the conditions specified would likely produce a material suitable for flotation feed. The data in Table II for the 20 minute ball mill grinds indicate that this grinding is quite reproducible.

The sample which was ground for 45 minutes was analysed for \( \text{U}_3\text{O}_8 \) by the thyrode method.* The \( \delta \) assay indicated 0.066% \( \text{U}_3\text{O}_8 \) compared to 0.0795% \( \text{U}_3\text{O}_8 \) indicated by the \( \beta \)-assay. The calculated \( \delta/\beta \) ratio was equal to 0.0624. The \( \delta/\beta \) ratio calculated for 100% equilibrium is 0.0456. These figures correspond to a secular equilibrium

* Lapointe, C. *Equilibrium Correction In Geiger Analysis.* Mines
The $\gamma/\beta$ ratio is the ratio of the $\gamma$ counting rate to the $\beta$ counting rate, these rates having been converted to counts per minute per 100 grams per one per cent $\text{U}_3\text{O}_8$. The $\gamma/\beta$ ratio for 100% equilibrium indicated above applies only to the thyrode tube used in these experiments.
SCREEN ANALYSES OF PRODUCTS OF VARIOUS SIZE REDUCTION OPERATIONS

Cumulative Mass Percent Passing Screen

45 min. ball-milling

20 min. ball-milling

after 2
passes
through
crushing
rolls

Screen Aperture (in.)
over 100%, indicating the possible presence of thorium in the ore. The equation

\[ c = 2.5B - 1.5G \]

where \( c \) = corrected \( \% \) \( \text{U}_3\text{O}_8 \)

\( B = \% \text{U}_3\text{O}_8 \) by the \( \beta-\gamma \) count

\( G = \% \text{U}_3\text{O}_8 \) by the \( \delta \) count

may be used to correct the thyrode assay\(^{(44)}\).

Applying the equation to the preceding results gave a corrected assay of 0.069 \( \text{U}_3\text{O}_8 \). Thompson \(^{(59)}\) using the tray method reported 0.074\% \( \text{U}_3\text{O}_8 \) for the same sample. In a later section of this work a comparison of analyses by the tray method and a fluorophotometric method is made. Two samples which analyzed 0.071\% \( \text{U}_3\text{O}_8 \) and 0.074\% \( \text{U}_3\text{O}_8 \), respectively, by the tray method, analyzed 0.067\% \( \text{U}_3\text{O}_8 \) fluorophotometrically. The fluorophotometric analysis is in excellent agreement with the analysis obtained by correcting the thyrode assay.

During the course of this work it was noticed that the analysis of the feed to the flotation cell varied somewhat from one test to another. In one bag of sample DES55-1 the analysis of the feed varied from 0.098\% to 0.066\% \( \text{U}_3\text{O}_8 \). Generally results near the higher figure were obtained with ore from near the bottom of the bag. The usual feed analysis was 0.074\% \( \text{U}_3\text{O}_8 \). While the variations in the feed analyses may be partially attributed to uncertainties in the analytical procedure, it seems likely that some classification is occurring within the bag. Due to its high density and small grain size the \( \text{U}_3\text{O}_8 \) would tend to concentrate at the bottom.
B. Assay Technique.

The method of analysis was the same as that used by previous workers on this project (1, 2, 3, 4, 59). It is discussed in some detail by Bulani (42). The material to be analyzed was given no size reduction treatment other than that received in the normal flotation procedure. Approximately one gram of the material to be analyzed was compressed into a small aluminum dish. This dish was then placed under an end-window Geiger tube and the number of registers in ten minutes was recorded. After rotating the sample through approximately 180° relative to the Geiger tube, the number of registers in 5 minutes was recorded. This rotation was done in an attempt to allow for any irregularities in compressing the sample in the tray. The arithmetic average of the two registering rates, corrected for background, was used in subsequent calculations. The specific registering rate for various weights of sample was then calculated. A straight line was obtained when the logarithm of the specific registering rate was plotted against the weight of the sample in grams (See Fig. 5). Using samples of known U₃O₈ content the specific registering rate at zero weight was related to the percentage U₃O₈ in the sample. On arithmetic graph paper the specific registering rate at zero weight plotted against % U₃O₈ gave a straight line passing through the origin (Fig. 6). The specific registering rate at zero weight for the sample being analyzed was obtained by extrapolating as indicated in Fig. 5 by the dashed lines. The % U₃O₈ was then read from a curve of the type shown in Fig. 6. This calibration curve was checked every two or three months. Only once was it necessary to alter the curve. The use of the specific registering rate at zero weight provides a correction for the absorption of β particles by the sample. In this connection it should be noted that Bulani (42) recommends that
TYPICAL GRAPH OF SPECIFIC REGISTERING RATE vs WEIGHT OF SAMPLE

Fig. 5

Specific Registering Rate

Tailings

Slimes

Concentrate

Feed

Weight of Sample (grams)

0.2 0.4 0.6 0.8 1.0 1.2 1.4 1.6 1.8 2.0
TYPICAL CALIBRATION CURVE
FOR END-WINDOW GEIGER TUBE

Fig. 6
the size of the sample used in the counting be not less than 0.6 grams for material ground to -200 mesh. In the case of samples of less than 0.6 grams, lines of steeper slope are noted when the logarithm of the specific registering rate is plotted against sample weight. Samples smaller than 0.6 grams could be used if the calibration were carried out with samples of similar size.

In the present work no attempt was made to correct the analyses for either the lack of secular equilibrium or the possible presence of thorium. It is not likely that the thorium would become separated from the uranium either in crushing or in flotation (45). Talbot concluded that the secular equilibrium of the ore was not affected by the flotation operation (1). The products of two flotation tests were analyzed by both the tray method previously described and a fluorophotometric method. The comparative results are given in Table I. Consideration of the data in this table shows that the results of the two analytical methods are in fair agreement. As this investigation is concerned primarily with obtaining high recoveries of uraninite in the concentrate, the application of corrections for lack of equilibrium and the presence of thorium is probably not necessary.

* The author is indebted to Dr. B. Gunn for the fluorophotometric analyses.

† Zimmermann, J. B. Department of Mines and Technical Surveys. Memorandum Series No. 115 (1951)
Table IIA. Comparative Analyses by the Tray Method and by the Fluorophotometric Method.

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Grade by Tray Method (% ( \text{U}_3\text{O}_8 ))</th>
<th>Grade by Fluorophotometric Method (% ( \text{U}_3\text{O}_8 ))</th>
<th>Weight Feed</th>
<th>% Recovery of the ( \text{U}_3\text{O}_8 ) in the Feed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>0.074</td>
<td>0.067</td>
<td>100.0</td>
<td>100.0</td>
</tr>
<tr>
<td>Slimes</td>
<td>0.215</td>
<td>0.19</td>
<td>2.3</td>
<td>6.6</td>
</tr>
<tr>
<td>Concentrate</td>
<td>0.13</td>
<td>0.12</td>
<td>15.1</td>
<td>25.4</td>
</tr>
<tr>
<td>Tailings</td>
<td>0.053</td>
<td>0.051</td>
<td>81.5</td>
<td>58.3</td>
</tr>
</tbody>
</table>

Test 100

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Grade by Tray Method (% ( \text{U}_3\text{O}_8 ))</th>
<th>Grade by Fluorophotometric Method (% ( \text{U}_3\text{O}_8 ))</th>
<th>Weight Feed</th>
<th>% Recovery of the ( \text{U}_3\text{O}_8 ) in the Feed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>0.071</td>
<td>0.067</td>
<td>100.0</td>
<td>100.0</td>
</tr>
<tr>
<td>Slimes</td>
<td>0.20</td>
<td>0.16</td>
<td>1.1</td>
<td>3.0</td>
</tr>
<tr>
<td>Concentrate</td>
<td>0.14</td>
<td>0.12</td>
<td>5.5</td>
<td>6.5</td>
</tr>
<tr>
<td>Tailings</td>
<td>0.065</td>
<td>0.063</td>
<td>94.1</td>
<td>88.2</td>
</tr>
</tbody>
</table>

Test 9 Table XI

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Grade by Tray Method (% ( \text{U}_3\text{O}_8 ))</th>
<th>Grade by Fluorophotometric Method (% ( \text{U}_3\text{O}_8 ))</th>
<th>Weight Feed</th>
<th>% Recovery of the ( \text{U}_3\text{O}_8 ) in the Feed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>0.071</td>
<td>0.067</td>
<td>100.0</td>
<td>100.0</td>
</tr>
<tr>
<td>Slimes</td>
<td>0.20</td>
<td>0.16</td>
<td>1.1</td>
<td>3.0</td>
</tr>
<tr>
<td>Concentrate</td>
<td>0.14</td>
<td>0.12</td>
<td>5.5</td>
<td>6.5</td>
</tr>
<tr>
<td>Tailings</td>
<td>0.065</td>
<td>0.063</td>
<td>94.1</td>
<td>88.2</td>
</tr>
</tbody>
</table>

* This designation refers to the author's laboratory records. This test is not referred to elsewhere in this work.

C. Contact Angle Measurements.

The importance of the contact angle in the flotation operation has been discussed in a previous section of this work. A "bubble machine" similar to that described by Taggart (15, p. 19-182) was erected and was used to determine the contact angles on some of the constituents of the ore after treatment with various flotation reagents. The procedure adopted in measuring the contact angles was similar to that used
by Talbot (1) and Korchinski (2). Mineral samples were polished with 4/0 emery paper, rinsed in distilled water, and placed in the conditioning medium for a specified length of time. After being removed from the conditioning medium and rinsed in a stream of distilled water, the samples were placed under distilled water in the glass cell of the "bubble machine." A small air bubble was brought to the surface of the solid by means of a fine capillary tube. The image of the bubble was magnified through a microscope and focused on a ground glass screen where the contact angle was measured. Before a particular reagent was investigated a "blank" was carried out on an untreated sample of the mineral. If the bubble adhered to this untreated surface, the mineral was further polished until the bubble showed no tendency to stick. The mineral sample "cleaned" in this fashion was treated with collector in the manner previously described. The results of these contact angle measurements are given in Tables III and IV. In this work a zero angle of contact means that the bubble would not adhere to the surface after the capillary tube was removed. Occasionally a bubble was observed which showed some tendency to stick, but would not remain on the surface when the capillary was removed. The contact angle on paraffin measured by the method described was 104° which agrees very well with the 105° reported by Adam (15). The tests reported in Table III lead to the following general conclusions:

(a) Compounds containing more than one polar group did not give a finite contact angle on uraninite. This effect may be due to the extreme insolubility in water of these reagents.

(b) Cyanamid reagent 712 showed promise of being a suitable collector for uraninite. This conclusion is corroborated by the results of flotation experiments.

Details of this procedure are given in Appendix B.
### Table III - Contact Angles on Uraninite after Treatment with Various Reagents.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Observed Contact Angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 min. conditioning in a 0.26% by weight mixture of 1-decanethiol in distilled water</td>
<td>48.0° (mean of 9 measurements)</td>
</tr>
<tr>
<td>20 min. conditioning in 1-decanethiol as supplied</td>
<td>50.0° (mean of 8 measurements)</td>
</tr>
<tr>
<td>10 min. conditioning in sat. soln. of 2-mercapto benzothiazol</td>
<td>0.0°</td>
</tr>
<tr>
<td>10 min. conditioning in Antarox A-40° as supplied</td>
<td>0.0°</td>
</tr>
<tr>
<td>10 min. conditioning in sat. soln. of Emulphor ON°</td>
<td>0.0°</td>
</tr>
<tr>
<td>10 min. conditioning in sat. soln. of α-mercapto-N-2-paphthyl acetamine</td>
<td>0.0°</td>
</tr>
<tr>
<td>20 min. conditioning in Cyanamid Reagent 712° as supplied</td>
<td>76.0° (mean of 7 measurements)</td>
</tr>
<tr>
<td>10 min. conditioning in a 1% soln. of Cyanamid Reagent 712</td>
<td>74° (mean of 4 measurements)</td>
</tr>
<tr>
<td>20 min. conditioning in a sat. soln. of α-mercapto-acetanilide</td>
<td>9°</td>
</tr>
<tr>
<td>20 min. conditioning in α-toluenethiol as supplied</td>
<td>0°</td>
</tr>
<tr>
<td>10 min. conditioning in a 0.26% by weight mixture of m-toluenethiol in distilled water</td>
<td>52.1° (mean of 5 measurements)</td>
</tr>
</tbody>
</table>

* A product of Chemical Developments of Canada Limited, Montreal and Toronto.

+ A product of the American Cyanamid Company, New York.
Table IV  Contact Angles on Various Constituents of the Ore

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Angle measured after 20 min. conditioning in Reagent 712 as supplied</th>
<th>Angle measured after 10 min. conditioning in a 1% soln. of Reagent 712</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uraninite</td>
<td>76° (mean of 7 measurements)</td>
<td>74° (mean of 4 measurements)</td>
</tr>
<tr>
<td>Labradorite</td>
<td>61°</td>
<td>0°</td>
</tr>
<tr>
<td>Oligoclase</td>
<td>51°</td>
<td>0°</td>
</tr>
<tr>
<td>Orthoclase</td>
<td>0°</td>
<td>0°</td>
</tr>
<tr>
<td>Biotite</td>
<td>75°</td>
<td>0°</td>
</tr>
<tr>
<td>Quartz</td>
<td>62°</td>
<td>61° (mean of 8 measurements)</td>
</tr>
</tbody>
</table>

In view of the large contact angle observed on uraninite following treatment with Cyanamid Reagent 712; the effect of this reagent on some of the gangue minerals was investigated. The results are shown in Table IV. It was noticed that the value of the contact angle depended greatly on the method of conditioning. Probably the results obtained after conditioning in the 1% solution more closely approximate conditions existing in a flotation cell. If the results reported in Column 3 of Table IV are taken as the more reliable, quartz seems to be the only gangue mineral likely to interfere in the flotation of uraninite with Cyanamid Reagent 712 as collector.
D. Flotation Tests Carried Out with the 50 Gram Denver Sub-A Flotation Cell.

An excellent sketch of the 50 gram flotation cell is given by Talbot (1). These machines consist essentially of a small "tank" mounted beneath a two speed electric motor. The motor carries a shaft and impeller extending to within approximately 1 inch of the bottom of the "tank" or cell. These machines may be operated as either agitation cells or sub-aeration cells. In the past these cells have been used for experiments of a qualitative nature (1, 2, 3, 4). Since it was intended to do work of a more quantitative nature, it was desirable to determine the reproducibility of the tests carried out with these small cells.

With the exception of the data reported in Table VII, the following series of tests were made on ore DE52-2.

In one series, eleven tests were conducted under the following conditions:

- Mass of sample - 50 grams.
- Pulp density - 20% dry solids by weight.
- Medium - a 4.75% by volume solution of absolute ethanol in distilled water.
- Mixing period at slow speed with no reagent - 10 minutes.
- Conditioning period at slow speed with 2.0 lb. of 1-demanthiol per per ton of ore - 20 minutes.
- Conditioning period at high speed 2.5 lb. of pine oil (frother) per per ton of ore - approximately 1 minute.
- Volume of concentrate collected at high speed - 250 ml.
- Time of collection at high speed - approximately 15 minutes.

The end point in these flotations was not determined by the character of the froth. In all these tests the same volume of concentrate was collected in approximately equal times. The variations in the grade of the concentrate and in the recovery are shown in Table VI.
In further experiments Thompson (39) attempted to duplicate a series of tests reported by the present author (5). The conditions for these experiments were similar to those outlined in the preceding paragraph with the following exceptions.

**Table V. Results of Replicate Experiments Conducted with the 50 Gram Denver Sub-A Flotation Cell.**

<table>
<thead>
<tr>
<th>Concentrate Grade % U₃O₈</th>
<th>% of Total U₃O₈ Recovered in Concentrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.22</td>
<td>36.0</td>
</tr>
<tr>
<td>0.10</td>
<td>25.5</td>
</tr>
<tr>
<td>0.11</td>
<td>26.9</td>
</tr>
<tr>
<td>0.12</td>
<td>20.0</td>
</tr>
<tr>
<td>0.12</td>
<td>17.4</td>
</tr>
<tr>
<td>0.10</td>
<td>14.1</td>
</tr>
<tr>
<td>0.32</td>
<td>78.0</td>
</tr>
<tr>
<td>0.15</td>
<td>23.1</td>
</tr>
<tr>
<td>0.25</td>
<td>84.5</td>
</tr>
<tr>
<td>0.33</td>
<td>55.3</td>
</tr>
<tr>
<td>0.34</td>
<td>56.1</td>
</tr>
</tbody>
</table>

These experiments were conducted on ore DE52-2 assaying 0.956% U₃O₈.

The amounts of reagents used by Thompson were: l-decanethiol, 1.04 lb./ton and pine oil, 1.58 lb./ton. In the previous work (5) by the present author, the amount of l-decanethiol was 0.96 lbs./ton and the amount of pine oil was 1.2 lb./ton. Thompson collected the concentrate for 20 minutes whereas the author carried collection to the point where the froth appeared barren. This occurred in 20 to 30 minutes. Thompson's data are reported here in Table VI, while those of the present
author are given in Table VII. The data contained in Tables VI and VII are shown graphically in Fig. 7.

Statistical calculations were made on the data contained in Table V. The concentrate grade showed a mean of 0.196% U₃O₈ and a standard deviation of 0.092% U₃O₈. The 95% confidence limits for the mean of the "population" from which this "sample" was drawn are 0.196±0.066% U₃O₈.

<table>
<thead>
<tr>
<th>% Ethanol by volume</th>
<th>Concentrate Grade</th>
<th>% of Total U₃O₈ recovered in concentrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.24</td>
<td>50.6</td>
</tr>
<tr>
<td>0</td>
<td>0.15</td>
<td>22.0</td>
</tr>
<tr>
<td>0</td>
<td>0.17</td>
<td>58.6</td>
</tr>
<tr>
<td>0</td>
<td>0.18</td>
<td>54.4</td>
</tr>
<tr>
<td>9.5</td>
<td>0.15</td>
<td>27.9</td>
</tr>
<tr>
<td>9.5</td>
<td>0.15</td>
<td>41.1</td>
</tr>
<tr>
<td>9.5</td>
<td>0.10</td>
<td>27.2</td>
</tr>
<tr>
<td>19.0</td>
<td>0.11</td>
<td>17.1</td>
</tr>
<tr>
<td>19.0</td>
<td>0.14</td>
<td>15.9</td>
</tr>
<tr>
<td>28.5</td>
<td>0.10</td>
<td>18.9</td>
</tr>
<tr>
<td>28.5</td>
<td>0.18</td>
<td>36.4</td>
</tr>
<tr>
<td>28.5</td>
<td>0.15</td>
<td>42.9</td>
</tr>
<tr>
<td>38.0</td>
<td>0.11</td>
<td>22.0</td>
</tr>
<tr>
<td>38.0</td>
<td>0.20</td>
<td>55.5</td>
</tr>
<tr>
<td>38.0</td>
<td>0.18</td>
<td>44.0</td>
</tr>
<tr>
<td>47.5</td>
<td>0.09</td>
<td>23.2</td>
</tr>
<tr>
<td>47.5</td>
<td>0.11</td>
<td>18.8</td>
</tr>
<tr>
<td>47.5</td>
<td>0.14</td>
<td>38.3</td>
</tr>
<tr>
<td>61.6</td>
<td>0.10</td>
<td>21.5</td>
</tr>
<tr>
<td>61.6</td>
<td>0.21</td>
<td>35.0</td>
</tr>
<tr>
<td>76.0</td>
<td>0.08</td>
<td>25.2</td>
</tr>
<tr>
<td>76.0</td>
<td>0.11</td>
<td>24.7</td>
</tr>
</tbody>
</table>

These experiments were conducted on ore DE 52-2 assaying 0.056% U₃O₈ (4).
Table VII - Effect of Ethanol Concentration on the Concentrate Grade and on the Recovery (3)

<table>
<thead>
<tr>
<th>% Ethanol by Volume</th>
<th>Concentrate Grade % U₃O₈</th>
<th>% of Total U₃O₈ recovered in concentrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.27</td>
<td>15.6</td>
</tr>
<tr>
<td>9.5</td>
<td>0.29</td>
<td>33.7</td>
</tr>
<tr>
<td>19.0</td>
<td>0.27</td>
<td>51.5</td>
</tr>
<tr>
<td>28.5</td>
<td>0.17</td>
<td>55.0</td>
</tr>
<tr>
<td>47.5</td>
<td>0.11</td>
<td>69.2</td>
</tr>
<tr>
<td>47.5</td>
<td>0.22</td>
<td>69.7</td>
</tr>
<tr>
<td>61.6</td>
<td>0.21</td>
<td>59.7</td>
</tr>
<tr>
<td>76.0</td>
<td>0.26</td>
<td>53.2</td>
</tr>
</tbody>
</table>

These experiments were conducted on ore DE 52-1 which analyzed 0.045% U₃O₈ (3)

The mean recovery was 39.5% with a standard deviation of 25.0%. The 95% confidence limits for the mean of the "population" of recoveries were 39.5±17.8%. In this discussion, "population" is used in the usual statistical sense. It is the total number of tests which could be conducted under the given set of conditions. In this case the population is infinite. The use of 95% confidence limits means that one can state with 95% confidence that the mean of the population falls within the given limits.

The data presented in Fig. 7 and in the results of the statistical calculation just given showed that the 50 gram cell was unsuitable for quantitative work, at least when it was used as an agitation cell. Possibly careful study would reveal the factors causing the deviations. It is interesting to note that authorities such as those at
VARIATION OF RECOVERY WITH CONCENTRATION OF ETHANOL USING 50 GRAM DENVER SUB-A FLOTATION CELLS

- O Author's data
- O Thompson's data

Fig. 7
the Mines Branch, Ottawa, believe cells smaller than 500 grams to be very unreliable (44).

E. Flotation Tests Carried Out with the 500 Gram Denver Sub-A Flotation Cell.

1. Description of Apparatus.

When it was decided to carry out more quantitative flotation work than was possible with the 50 gram cells, a 500 gram Denver Sub-A flotation cell was put into operation. A photograph of this cell is shown in Fig. 8. The rotor of this cell is belt-driven from a \( \frac{1}{2} \) H.P., variable speed electric motor. The rotor is surrounded by a stand-pipe equipped with a valve at the top. Through this stand-pipe air is delivered directly to the impeller and dispersed throughout the pulp. The valve may be used to control the amount of air being admitted.

The actual flotation cell consists of two zones. In the rear compartment (the agitation zone), the pulp is mixed thoroughly with air and reagents. The pulp then flows up over a dividing wall and into the separating zone where the mineral laden froth is removed. The remaining material flows down, under the dividing partition, and back into the agitation zone. In the flotation cell used in this work, the froth was removed automatically by a paddle rotating at approximately 50 r.p.m.

Through a system of belts and pulleys this paddle is driven by an electric motor rotating at 1450 r.p.m. The small spigot on the side of the cell was used for the removal of a sample for pH and pulp density determinations in each test.

The ore samples were prepared for flotation by wet grinding in a 12 in. by 5 in. Denver laboratory ball mill. The procedure followed for the grinding has been described in Section IV, Sub-section A, of this
Photograph of 500 Gram Flotation Cell

Fig. 8
work. The Denver laboratory ball mill is driven by an electric motor, through a gear box and a belt and pulley system. Several sizes of pulley are available in order that the speed may be varied. In this work the speed was always 54 r.p.m.

2. Development of the Flotation Test Procedure.

The change from the 50 gram flotation cells to the 500 gram cell was accompanied by a considerable alteration in the test procedure. The procedure finally adopted was the result of several weeks' work during which experience in handling the apparatus was gained. Some of the difficulties encountered will be reviewed here in an attempt to show how the procedure developed. It is hoped that the information will be useful to others carrying on this research.

In the first two or three tests carried out with the large cell the ore charge was fed into the forward or separating compartment of the cell. This practice led to considerable difficulty in discharging the tailings from the cell following the test. In subsequent tests, when the ore was fed directly into the agitation zone, no such plugging occurred.

When the change from the 50 gram cells to the 500 gram cells occurred, a desliming step was added to the flotation test procedure. One test was carried out in which the ore was deslimed right in the flotation cell. The original ore charge was ground for 20 minutes under the conditions previously specified. Three fractions of slimes were removed, the pulp being raised to the original level between each fraction. No determination of pulp density was made. The three agitation periods were each of five minutes duration. The rotor was run at 1500 revolutions per minute. Settling periods of three, three, and four minutes, respectively, were allowed. As much as possible of the supernatant suspension
was removed by means of a siphon. The slime fraction contained 35.6% of the \( \text{U}_3\text{O}_8 \) at a grade of 0.108\% \( \text{U}_3\text{O}_8 \). The original feed analyzed 0.074\% \( \text{U}_3\text{O}_8 \). This desliming procedure was found to be very unsatisfactory, mainly due to the fact that it was difficult to gauge the depth of the siphon accurately.

Subsequent desliming of the feed to the flotation cell was carried out in a four-litre beaker at a pulp density of approximately 18.0\%. The agitator was driven by a small electric motor. The agitator reached to within approximately 1.5 in. of the bottom of the vessel. A few tests were carried out in which practically all the supernatant suspension was removed by means of a siphon, the tip of which was directed vertically downward. During the course of these tests the lengths of the agitation period and of settling period were varied. The length of the agitation period did not seem to be critical, provided sufficient time was allowed to get most of the material into suspension. The time required appeared to be less than 2 minutes. As might be expected, the results of the desliming operation depend greatly on the length of the settling period. As before, three replicate periods of agitation and settling were allowed. After each settling period the suspended material was removed by means of a siphon. In one instance, when the agitation periods and the settling periods were two minutes and five minutes, respectively, the slimes contained 57.5\% of the \( \text{U}_3\text{O}_8 \) at a grade of 0.12\% \( \text{U}_3\text{O}_8 \) from a feed analyzing 0.074\% \( \text{U}_3\text{O}_8 \). Recoveries at high as 40\% in the slimes were quite common. It was thought that the removal of such large percentages of the \( \text{U}_3\text{O}_8 \) would make the results of the actual flotation experiments.

The next modification in the desliming procedure was the removal of only the top 15 cm. of slurry. Again the siphon used had the tip directed vertically downward. This modification did not yield results
significantly different from those obtained by removing practically all the supernatant suspension. It was also noticed that this desliming procedure lacked reproducibility. Recoveries ranging from 24.2% to 48.9% of the \( \text{U}_3\text{O}_8 \) were obtained in the slimes by this method when the settling periods were ten minutes and the agitation periods were two minutes. In the desliming procedure discussed in this paragraph and in the preceding one, it was common to remove from 25% to 40% of the original ore charge in the slime fraction. Examination of the screen analysis of the material ground for 20 minutes in the ball mill (Fig. 4) shows that removal of this percentage of the ore corresponds to almost complete removal of the -400 mesh fraction, whereas slimes are usually considered to be material much finer than this (5, p. 12-21).

The desliming procedure finally adopted consisted of agitating the pulp, at a pulp density of approximately 18% for two minutes, allowing a 10-minute settling period, and removing the top 10 cm. of slurry. This procedure was carried out three times. The slurry was removed by means of a siphon, the tip of which was reversed so that it pointed towards the top of the vessel. The use of such a siphon allows the removal of the suspension without agitating the material which has settled.

It is interesting to note that recoveries of the order of 50% of the \( \text{U}_3\text{O}_8 \) were obtained by simply allowing the coarse fraction of the ore to settle and siphoning off the suspended material. Thompson (59), in a further investigation into the desliming procedure, obtained a recovery of 95.5% of the \( \text{U}_3\text{O}_8 \) at a grade of 0.11% \( \text{U}_3\text{O}_8 \) by agitating the pulp for two minutes, allowing no settling period before starting to siphon, and removing the top 15 cm. of slurry. This test was carried out on material which had been ground in the ball mill for 20 minutes.
The feed assayed approximately 0.074\% \text{U}_3\text{O}_8. (The feed to every test was not analyzed at this point in the work). In connection with this high recovery obtained by Thompson, it should be pointed out that the \text{U}_3\text{O}_8 accounted for in the various products of the operation was 118.0\% of the \text{U}_3\text{O}_8 in the feed.

Unsatisfactory \text{U}_3\text{O}_8 balances led to the conclusion that the analysis of the feed was varying from one test to another. A possible reason for this variation has been discussed in Section IV, Sub-section A of this work. It was decided to conduct an analysis of the feed and to calculate overall material and \text{U}_3\text{O}_8 balances for every test. The flotation test procedure finally adopted involved the following steps. The sample weighing 700 grams was placed in the ball mill with 350 ml. of distilled water, 5 lb. each of 0.75 in. nominal diameter steel balls, 1.0 in. nominal diameter steel balls, and 1.5 in. nominal diameter steel balls. The ball mill was then rotated at 54 r.p.m. for the desired time. This grinding time was usually 20 minutes and in the reporting of subsequent flotation experiments it may be taken as such unless otherwise stated. The product of this size reduction operation was washed into a pail with distilled water and a sample taken for feed analysis. In the majority of the tests this sample was taken by filtering the product of the ball mill and cutting a wedge of approximately 20 grams from the wet cake. On the advice of workers at the Mines Branch, Ottawa, this sampling procedure was changed, and in a few of the tests the feed sample was taken by simply thoroughly agitating the ground material in the pail into which it was washed from the mill and dipping out 100 ml. of pulp. The analyses of the feed samples obtained by this method did not seem to differ significantly from the feed analyses obtained when the sampling procedure
involved filtration of the ball mill product. No deviations in the $\text{U}_3\text{O}_8$ balances calculated from the feed analysis & obtained by these two sampling methods were noted. After removal of the feed sample, the ball mill product was washed into a four-litre beaker and brought to a pulp density between 17.5 and 18.0% dry solids by weight with distilled water. Desliming was then carried out in three stages. In each stage the duration of the agitation period was two minutes, and the duration of the settling period was ten minutes. The pulp was brought to the original dilution with distilled water following each desliming step. The material remaining in the desliming vessel after the third desliming stage was washed into the flotation cell and made up to the desired pulp density with distilled water. Any modifying agents used in the flotation were then added and the pulp agitated at 1500 r.p.m. for a specified length of time, usually ten minutes. Following this "preconditioning period" the pulp was conditioned with the collector for a specified time, usually ten minutes. Half way through this "conditioning period" a 10 ml sample of pulp was taken for pH and pulp density determinations. During both the preconditioning period and the conditioning period the air valve was closed. The speed of the agitator was increased to its full value\* following the conditioning period and the frother was added. The pulp was conditioned with the frother for three minutes before the air valve was opened and collection started. The concentrate was collected for the desired length of time or until the character of the froth indicated the end point had been reached. During the course of the flotation, distilled water was added intermittently to the rear compartment to replace that carried over in the froth. In a few tests the

\*The top speed was 2100 r.p.m. with the air valve closed and 2200 r.p.m. with the air valve open
frother was added periodically during the collection period. Following the flotation, the tailings were discharged from the cell. The three fractions, slimes, concentrate and tailings, were filtered, dried and analysed for $U_3O_8$ by the tray method. A total material balance and a $U_3O_8$ balance were calculated for each test. The flotation cell was cleaned after each test by being rinsed with distilled water while the impeller was in motion.

3. Results of Flotation Experiments.

The results of flotation experiments to be reported in this work will be classified on the basis of the collector used. In a few instances some departure from this classification will be necessary. Some of the modifying agents investigated were tested with various collectors. No thorough investigation of the reproducibility of the experiments carried out with the 500 gram cell was made. Attention is called to the three tests conducted at a pH of 10.7 and the one test at a pH of 10.8 which are reported in Table VIII. The results of these experiments indicate that the reproducibility of the experiments carried out with the 500 gram cell is considerably better than was obtained with the 50 gram cells.

(a) Thioalcohols as Collectors.

Korchinski reported (2) that certain thioalcohols exhibited selectivity towards uraninite in flotation. Craig (4) and the present author (3) also investigated the use of thioalcohols as collectors for uraninite. In most of these experiments the flotation media were solutions of ethanol in water. While results obtained under these conditions are of interest, the cost of such operations on a commercial scale would probably be prohibitive. In the present work it was decided to investig-
gate the flotation of uraninite with thioalcohols as collectors in a water medium.

The first series of experiments was conducted to determine the effect of the number of carbon atoms in the mercaptan on the recovery and on the grade of the concentrate obtained. The experimental procedure as previously outlined was followed. The amount of collector used was approximately 0.89 lb./ton, based on the amount of ore actually in the cell. Approximately 0.24 lb. of Dowfroth 250* per ton of ore was used as frother. The reagents were added by means of medicine droppers. There were some slight variations in the amounts of reagents present. These variations were due to differences in the amounts of ore in the cell, and to differences in the densities of the collectors. The variations in the amount of ore in the cell were due to the fact that it was impossible to exactly duplicate the size of sample taken for feed analysis. It was originally intended to carry out these tests in a neutral medium. Several runs were made under this condition, but no recoverable froth formed. When the medium was made strongly alkaline (pH 11.5) with sodium hydroxide, a froth formed which was quite deep and stable. It is interesting to note that in a neutral medium with no mercaptan present a stable froth formed.

In the experiments reported here the pH was brought to 11.5 during the conditioning period by means of sodium hydroxide, and the pulp density was between 25% and 28% dry solids by weight. The concentrate was collected for 10 minutes. The end point was not determined by the character of the froth. However, after 10 minutes the froth was usually barren. The effect of the number of carbon atoms in the mercaptan on the recovery and on the concentrate grade is shown in Table

* A product of the Dow Chemical Company, San Francisco, California.
VII and graphically in Fig. 9. There does not seem to be any significant variation in either the recovery or the grade of the concentrate.

Table VII - Effect of Number of Carbon Atoms in Mercaptan on Recovery and Concentrate Grade.

<table>
<thead>
<tr>
<th>Collector Molecule</th>
<th>No. of C Atoms per Analysis</th>
<th>Feed Slimes Grade %U₃O₈</th>
<th>Recovery Grade %U₃O₈</th>
<th>Concentrate Grade %U₃O₈</th>
<th>Tailings Grade %U₃O₈</th>
</tr>
</thead>
<tbody>
<tr>
<td>None</td>
<td>0</td>
<td>0.071</td>
<td>0.18</td>
<td>4.6</td>
<td>0.11</td>
</tr>
<tr>
<td>I-propanethiol</td>
<td>3</td>
<td>0.075</td>
<td>0.17</td>
<td>0.6</td>
<td>0.16</td>
</tr>
<tr>
<td>I-hexanethiol*</td>
<td>6</td>
<td>0.067</td>
<td>0.19</td>
<td>5.9</td>
<td>0.15</td>
</tr>
<tr>
<td>I-octanethiol</td>
<td>8</td>
<td>0.065</td>
<td>0.18</td>
<td>3.3</td>
<td>0.14</td>
</tr>
<tr>
<td>I-decanethiol</td>
<td>10</td>
<td>0.076</td>
<td>0.23</td>
<td>9.1</td>
<td>0.17</td>
</tr>
</tbody>
</table>

* The U₃O₈ balance for this test is low.

In view of the fact that the addition of sodium hydroxide to the medium greatly enhanced the stability of the froth in the preceding series of experiments, a more thorough investigation of the effect of pH on the recovery and on the grade of the concentrate was carried out. In this series of experiments the collector was I-decanethiol and the frother was Dowfroth 250. The reagent consumption was: I-decanethiol, 0.89 lbs. per ton, and Dowfroth, 250, 0.24 lbs. per ton, based on the amount of ore in the cell. The procedure was similar to that previously outlined. The concentrate was collected for 5 minutes. At the end of this time the froth was usually barren. The pulp density was between 25% and 29% dry solids by weight. As in the previous series of experiments, a stable froth could not be obtained in the absence of sodium hydroxide. The results of these experiments are given in Table VIII, and the vari-
AFFECT OF NUMBER OF CARBON ATOMS IN MERCAPTAN ON RECOVERY AND ON THE GRADE OF THE CONCENTRATE

Fig.9
ations in the recovery and in the concentrate grade are shown graphically in Fig. 10. It will be noticed that the minimum grade occurs at the same pH as the maximum recovery. This is usual in flotation op-

Table VIII - Effect of pH on the Results of Flotation Experiments

in which 1-Decanethiol and Dowfroth 250 were used.

<table>
<thead>
<tr>
<th>pH</th>
<th>Feed Analysis</th>
<th>Slimes Grade</th>
<th>Slimes Recovery</th>
<th>Concentrate Grade</th>
<th>Concentrate Recovery</th>
<th>Tailings Grade</th>
<th>Tailings Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.0</td>
<td>0.076</td>
<td>0.22</td>
<td>5.2</td>
<td>0.24</td>
<td>21.7</td>
<td>0.064</td>
<td>75.8</td>
</tr>
<tr>
<td>7.6</td>
<td>0.080</td>
<td>0.22</td>
<td>6.4</td>
<td>0.22</td>
<td>8.2</td>
<td>0.074</td>
<td>87.5</td>
</tr>
<tr>
<td>8.8</td>
<td>0.075</td>
<td>0.22</td>
<td>8.5</td>
<td>0.26</td>
<td>18.4</td>
<td>0.060</td>
<td>70.4</td>
</tr>
<tr>
<td>9.9</td>
<td>0.075</td>
<td>0.20</td>
<td>4.3</td>
<td>0.22</td>
<td>22.2</td>
<td>0.065</td>
<td>77.0</td>
</tr>
<tr>
<td>9.9</td>
<td>0.080</td>
<td>0.21</td>
<td>10.3</td>
<td>0.42</td>
<td>19.2</td>
<td>0.063</td>
<td>70.0</td>
</tr>
<tr>
<td>10.7</td>
<td>0.078</td>
<td>0.22</td>
<td>6.4</td>
<td>0.20</td>
<td>34.5</td>
<td>0.060</td>
<td>62.5</td>
</tr>
<tr>
<td>10.7</td>
<td>0.072</td>
<td>0.22</td>
<td>2.0</td>
<td>0.21</td>
<td>38.7</td>
<td>0.052</td>
<td>60.9</td>
</tr>
<tr>
<td>10.8</td>
<td>0.074</td>
<td>0.21</td>
<td>7.4</td>
<td>0.19</td>
<td>38.2</td>
<td>0.052</td>
<td>57.9</td>
</tr>
<tr>
<td>10.7</td>
<td>0.074</td>
<td>0.21</td>
<td>2.5</td>
<td>0.17</td>
<td>39.6</td>
<td>0.047</td>
<td>50.1</td>
</tr>
<tr>
<td>11.7</td>
<td>0.074</td>
<td>0.20</td>
<td>8.2</td>
<td>0.22</td>
<td>18.0</td>
<td>0.059</td>
<td>69.4</td>
</tr>
</tbody>
</table>

* Sodium sulfide used as activator.
† 1-decanethiol emulsified with Emcol X-25.

Herations. At the point of maximum recovery the concentrate contains many particles consisting of large percentages of gangue minerals. Inclusion of these middling particles in the concentrate brings about a reduction in the concentrate grade. One experiment was conducted in which the concentration of Dowfroth 250 was 1.16 lb./ton. The frother was added intermittently during the collection period. The rate of addition of frother was governed by the appearance of the froth, enough being added to provide a stable, deep froth. The amount
EFFECT OF pH ON RECOVERY AND CONCENTRATE GRADE IN FLOTATIONS WITH 1-DECANETHIOL AND DOWFROTH 250

![Graph showing the effect of pH on recovery and concentrate grade in flotation using 1-decanethiol and Dowfroth 250.](image-url)

Fig. 10
of 1-decanethiol was 0.77 lb./ton. Inadvertently 800 grams of ore was used rather than the usual 700, so that the results of this experiment may not be strictly comparable with those obtained using 700 grams of ore. This experiment was conducted at a pH of 10.9 and a pulp density of 32.5%. In 10 minutes a concentrate containing 54.5% of the uranium values at a grade of 0.13% \( \text{U}_3\text{O}_8 \) was obtained. At the end of this collection period the froth was barren. The feed to this test analyzed 0.036% \( \text{U}_3\text{O}_8 \). The significance of the results of this test will be discussed in connection with the work done with Cyanamid Reagent 712.

In both Table VII and Table VIII it will be noticed that the grade of tailings was quite high and was maintained relatively constant throughout the series of experiments. Possible causes of this are poor contact between the collector and the uraninite or lack of liberation of the uraninite in the feed to the flotation cell. In an attempt to determine something of the character of the tailings a screen and radiometric analysis of accumulated tailings was performed. These tailings were accumulated from flotation experiments done in the large cell which are not reported in this work. Various collectors were used in these tests which were carried out during the development of the procedure for experiments in the 500 gram cell. The analyses of the tailings which made up this accumulated material were approximately the same as those reported in Tables VII and VIII. The data are presented in Table IX and graphically in Fig. 11. The \( \sqrt{2} \) Tyler standard screen series* was used in carrying out all screen analyses reported in this work.

* For an explanation of the terminology, see Ref. 6, pp.17-18.
### Table IX - Screen and Radiometric Analysis of Accumulated Tailings from Experiments with the 500 gram Flotation Cell.

<table>
<thead>
<tr>
<th>Screen Fraction</th>
<th>Weight % Retained</th>
<th>Average Particle size (in.)</th>
<th>% U₃O₈</th>
<th>% of Total U₃O₈</th>
</tr>
</thead>
<tbody>
<tr>
<td>-20 + 28</td>
<td>0.8</td>
<td>0.028</td>
<td>0.035</td>
<td>0.44</td>
</tr>
<tr>
<td>-28 + 55</td>
<td>0.8</td>
<td>0.020</td>
<td>0.030</td>
<td>0.37</td>
</tr>
<tr>
<td>-35 + 48</td>
<td>0.7</td>
<td>0.014</td>
<td>0.035</td>
<td>0.38</td>
</tr>
<tr>
<td>-48 + 65</td>
<td>2.7</td>
<td>0.010</td>
<td>0.036</td>
<td>1.50</td>
</tr>
<tr>
<td>-65 +100</td>
<td>11.0</td>
<td>0.007</td>
<td>0.030</td>
<td>5.02</td>
</tr>
<tr>
<td>-100 + 150</td>
<td>13.4</td>
<td>0.0039</td>
<td>0.030</td>
<td>6.11</td>
</tr>
<tr>
<td>-150 + 200</td>
<td>5.0</td>
<td>0.0035</td>
<td>0.030</td>
<td>2.28</td>
</tr>
<tr>
<td>-200 + 270</td>
<td>26.9</td>
<td>0.0025</td>
<td>0.060</td>
<td>24.45</td>
</tr>
<tr>
<td>-270 + 400</td>
<td>15.0</td>
<td>0.0018</td>
<td>0.075</td>
<td>17.00</td>
</tr>
<tr>
<td>-400</td>
<td>22.4</td>
<td>0.0007*</td>
<td>0.14</td>
<td>47.4</td>
</tr>
<tr>
<td></td>
<td>98.7</td>
<td></td>
<td></td>
<td>104.95</td>
</tr>
</tbody>
</table>

* This value was taken to be one half of the aperture of the 400 mesh screen.

Based on the amount of material fed to the screens: 400.0 gms.

Consideration of Table IX reveals that almost 80% of the U₃O₈ remaining in the tailings is in the -200 mesh fraction. This result would seem to indicate that lack of liberation was not the major factor causing the high tailings loss since Mawdsley (37) reports the uraninite size as approximately 0.2 mm. or 0.051 in., whereas the aperture of a 200 mesh screen is 0.074 mm. or 0.0029 in.

In an attempt to achieve better contact between the uraninite and the collector (1-decanethiol) an attempt was made to emulsify the collector using Emulol x 25 as the emulsifying agent. In one test the amount of 1-decanethiol was 0.89 lb./ton. This was emulsified with...
DISTRIBUTION OF $\text{U}_3\text{O}_8$ IN TAILINGS

ACCUMULATED FROM TESTS IN THE

500 GRAM DENVER SUB-A FLOTATION CELL

![Graph showing distribution of $\text{U}_3\text{O}_8$ in tailings]
an amount of Hacol X25 equivalent to 0.017 lb./ton by simply mixing the reagents with water in a test tube. The pH was brought to 10.8 with sodium hydroxide. The pulp density was 25.0%. A concentrate containing 38.2% of the uranium values at a grade of 0.19% U₃O₈ was obtained from a feed analyzing 0.074% U₃O₈. The collection period was 5 minutes at the end of which time the froth was practically barren.

The effect of activation by sodium sulfide with 1-decanethiol as collector was briefly investigated. In the one experiment performed the conditions were similar to those used to obtain the data in Table VIII. The amount of sodium sulfide used was 1.0 lb./ton. The pH was 10.7 and the pulp density was 25.0%. The concentrate collected in 5 minutes contained 38.7% of the uranium values at a grade of 0.21% U₃O₈. Neither this result nor that obtained using emulsified 1-decanethiol differ significantly from the results at this pH value reported in Table VIII.

In a further attempt to achieve better contact between the uraninite and 1-decanethiol an attempt was made to form a soluble salt of the mercaptan. A few ml. of 1-decanethiol were treated with a small piece of sodium metal for 1.5 hours at 75°C. During the reaction a yellowish white solid appeared in the bottom of the reaction vessel. The product was filtered, washed with ether and allowed to dry. A small portion of the material prepared was tested for solubility in water. The white solid disappeared with the formation of an oily layer at the surface of the water. This oily layer possibly consisted of 1-decanethiol released through a hydrolysis reaction. A similar reaction occurred in a solution of pH 10.5-11.0. In solutions more strongly alkaline than this the material was insoluble. A flotation test was conducted using this solid reaction product as collector. The amount of
collector was 0.89 lb./ton. The frother was pine oil, 0.24 lb./ton. The concentrate contained 18.3% of the U3O8 at a grade of 0.13% U3O8. The feed analyzed 0.074% U3O8. The pH was 7.3 and the pulp density was 27.5%.

In all the tests reported so far in this work, with the exception of the one in which Dowfroth 250 was added intermittently, it was noticed that the froth showed a tendency to deteriorate towards the end of the collection period. Following a suggestion of Mr. Gow of the Mines Branch, Ottawa, it was decided to investigate the characteristics of Cyanamid Reagent 712\textsuperscript{*} as a frother in the hope of obtaining stronger froths. In view of the large contact angle exhibited by Reagent 712 on uraninite, this reagent will be classed as a collector for the purpose of this work.

(b) Cyanamid Reagent 712 as a Collector and Frother.

Cyanamid Reagent 712 is described as a soap-type frother having certain promotor characteristics (45). The manufacturers claim that it produces a tough, non-selective froth useful in recovering locked middling particles. Subsequent work with Cyanamid Reagent 712 indicated that it did exhibit some selectivity towards uraninite. The experiments carried out with Cyanamid Reagent 712 are summarized in Table A.

The original intention was to use Cyanamid Reagent 712 as a frother. In Tests 1 to 5, Table A, 1-decanethiol was used as collector. Test 1 in Table A was carried out under the optimum conditions with regard to pH found in the experiments reported in Table VIII with the exception that Dowfroth 250 was replaced by Cyanamid Reagent 712. The recovery was significantly lower than that obtained using Dowfroth 250.

as frother under otherwise similar conditions. In this test the froth still showed a tendency to deteriorate towards the end of the flotation. It was decided to increase the concentration of Cyanamid Reagent 712 and to add this reagent in stages during the collection period so as to obtain a good froth throughout that period. At a pH of 10.1 a concentrate containing 54.9% of the uranium values at a grade of 0.20% U₃O₈ was obtained. (Test 4, Table X). Repetition of this experiment at a pH of 6.9 (Test 3, Table X) yielded a concentrate containing 78.9% of the uranium values at a grade of 0.12% U₃O₈. Addition of HCl to lower the pH to 6.6 (Test 5, Table X) caused a slight reduction in the recovery to 70.8%. The grade of the concentrate remained approximately the same as in Test 5 and was 0.13% U₃O₈. The character of the froths obtained in Tests 3, 4 and 5 is worthy of note. The froth formed in the neutral medium was fairly voluminous and consisted of relatively small bubbles. At a pH of 10.1 the froth was less voluminous but consisted of smaller bubbles. The most striking change was noted in the acid medium. The froth was still voluminous but in this instance consisted mainly of large bubbles. Some of these bubbles appeared to reach a size of 4 in. to 6 in. across.

The results of these experiments led to the conclusion that perhaps the concentration of Dowfroth 250 (0.24 lb./ton) had been too low in the previous series of experiments using 1-decanethiol as collector. It was at this point that the experiment using 1.16 lb. of Dowfroth 250/ton was performed (See p.61). It will be recalled that this test yielded a concentrate containing 54.5% of the U₃O₈ at a grade of 0.13% U₃O₈. While the recovery agrees favorably with Test 4, Table X, the grade is considerably lower than was obtained with Cyanamid Reagent
Table X - Summary of Experiments with Cyanamid Reagent 712.

<table>
<thead>
<tr>
<th>Collector</th>
<th>Lbs. of Reag.712 per ton</th>
<th>pH</th>
<th>Collection time (min.)</th>
<th>Pulp Density Analysis</th>
<th>Slimes % Recovery</th>
<th>Concentrate Grade % Recovery</th>
<th>Tailings Grade % Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) 1-decanethiol 0.30 lb/ton</td>
<td>0.44</td>
<td>10.3</td>
<td>5.5</td>
<td>25.6</td>
<td>0.096</td>
<td>0.22</td>
<td>3.1</td>
</tr>
<tr>
<td>2) 1-decanethiol 0.56 lb/ton</td>
<td>1.27</td>
<td>7</td>
<td>13</td>
<td>28</td>
<td>0.075</td>
<td>0.19</td>
<td>3.2</td>
</tr>
<tr>
<td>3) 1-decanethiol 0.89 lb/ton</td>
<td>1.32</td>
<td>6.9</td>
<td>13</td>
<td>26.1</td>
<td>0.081</td>
<td>0.19</td>
<td>1.1</td>
</tr>
<tr>
<td>4) 1-decanethiol 0.91 lb/ton</td>
<td>1.35</td>
<td>10.1</td>
<td>13</td>
<td>27.5</td>
<td>0.074</td>
<td>0.16</td>
<td>0.7</td>
</tr>
<tr>
<td>5) 1-decanethiol 0.89 lb/ton</td>
<td>1.51</td>
<td>6.6</td>
<td>13</td>
<td>27.5</td>
<td>0.074</td>
<td>0.16</td>
<td>0.5</td>
</tr>
<tr>
<td>6) None</td>
<td>1.32</td>
<td>7.5</td>
<td>13</td>
<td>28.5</td>
<td>0.777</td>
<td>0.16</td>
<td>0.8</td>
</tr>
<tr>
<td>7) None</td>
<td>1.30</td>
<td>6.7</td>
<td>13</td>
<td>28.5</td>
<td>0.078</td>
<td>0.18</td>
<td>0.6</td>
</tr>
<tr>
<td>8) None</td>
<td>1.30</td>
<td>4.7</td>
<td>13</td>
<td>27.5</td>
<td>0.074</td>
<td>0.20</td>
<td>4.0</td>
</tr>
<tr>
<td>9) None</td>
<td>1.31</td>
<td>11.7</td>
<td>13</td>
<td>28.5</td>
<td>0.071</td>
<td>0.20</td>
<td>3.0</td>
</tr>
</tbody>
</table>

† Ore roasted with sulfur prior to flotation

Note: Froth was collected until it became barren.

* Concentrate collected in six fractions.
712. The recovery is much higher than was obtained with smaller amounts of Dowfroth 250 in the thioalcohol experiments previously discussed. Test 2, Table X, was carried out on an ore charge that had been given pretreatment by roasting with sulfur. According to Taggart (5, p.12-10) thioalcohols are sulfide collectors. A 700 gram charge of ground ore was mixed with 1.149 grams of sulfur and roasted for 170 minutes at approximately 820°C. After being roasted the ore charge turned a reddish-copper color. The charge seemed to contain a considerable number of particles which had a definite metallic lustre. These particles were gold colored. The results obtained in this experiment do not appear to differ significantly from those obtained under similar conditions when the ore was not roasted with sulfur prior to flotation (Test 3, Table X).

In the tests of Table X in which recoveries of the order of 80% were obtained, the grade of the tailings was approximately 0.05% U₃O₈. This result corresponds to a significant reduction in the grade of the tailings when compared to the results presented in Table IX and Fig. 11. A screen and radiometric analysis of the tailings from Test 3 Table X, was conducted. The data are given in Table XI. The distribution of U₃O₈ in the tailings is shown graphically in Fig. 12. A comparison of this screen analysis with that previously given for accumulated tailings is interesting. It will be noticed that in the screen analysis presented in Table XI only 11.6% of the U₃O₈ remaining in the tailings is contained in the -400 mesh portion. In the screen analysis presented in Table IX 47.4% of the U₃O₈ lost in the tailings was in the -400 mesh portion. It would appear that in the experiment with Reagent 712 most of the U₃O₈ lost in the tailings was locked in the larger particles.
Table XI - Screen and Radiometric Analyses of Tailings

From Test 5, Table X.

Conditions: Collector, 1-decanethiol, 0.89 lb./ton  
Frother, Reagent 712, 1.32 lb./ton  
PH, 8.9

<table>
<thead>
<tr>
<th>Screen Fraction</th>
<th>Average Particle size, inches</th>
<th>Wt.% retained</th>
<th>%U₃O₈</th>
<th>% of total U₃O₈</th>
</tr>
</thead>
<tbody>
<tr>
<td>-14 + 20</td>
<td>0.0594</td>
<td>0.5</td>
<td>N.A.</td>
<td>-</td>
</tr>
<tr>
<td>-20 + 28</td>
<td>0.028</td>
<td>0.4</td>
<td>N.A.</td>
<td>-</td>
</tr>
<tr>
<td>-28 + 35</td>
<td>0.020</td>
<td>0.5</td>
<td>N.A.</td>
<td>-</td>
</tr>
<tr>
<td>-35 + 48</td>
<td>0.014</td>
<td>0.9</td>
<td>N.A.</td>
<td>-</td>
</tr>
<tr>
<td>-48 + 65</td>
<td>0.010</td>
<td>4.4</td>
<td>N.A.</td>
<td>-</td>
</tr>
<tr>
<td>-65 + 100</td>
<td>0.007</td>
<td>16.2</td>
<td>0.025</td>
<td>14.2</td>
</tr>
<tr>
<td>-100 + 150</td>
<td>0.0049</td>
<td>23.0</td>
<td>N.A.</td>
<td>-</td>
</tr>
<tr>
<td>-150 + 200</td>
<td>0.0055</td>
<td>17.9</td>
<td>0.051</td>
<td>19.4</td>
</tr>
<tr>
<td>-200 + 270</td>
<td>0.0025</td>
<td>15.6</td>
<td>0.036</td>
<td>19.7</td>
</tr>
<tr>
<td>-270 + 400</td>
<td>0.0018</td>
<td>11.0</td>
<td>N.A.</td>
<td>-</td>
</tr>
<tr>
<td>-400</td>
<td>0.0007*</td>
<td>4.8</td>
<td>0.067</td>
<td>11.6</td>
</tr>
</tbody>
</table>

100.0

N.A. means, "Not analyzed for U₃O₈."

* Taken as one half the aperture of the 400 mesh screen
++ Based on the sum of the weights of the fractions: 301.2 gms.
Weight of feed was 307.0 gms.

In an attempt to further investigate the effect of Cyanamid Reagent 712 on the flotation of uraninite a test was made in which no 1-decanethiol was used. The conditions for this test are given under Item 6 in Table X. The results obtained were not significantly different from those obtained, under similar conditions, with 1-decanethiol
Fig. 12

DISTRIBUTION OF U₃O₈ IN TAILINGS
FROM TEST CONDUCTED WITH CYANAMID
REAGENT 712 (Test 3, Table X)
present (Test 3, Table X). Obviously the mercaptan is not necessary under these conditions.

In Test 7, Table X, the concentrate was collected in six fractions. These fractions were analyzed separately and the data contained in Table XII were calculated. Fig. 15 was plotted by using the data in Table XII.

Table XII - Variation of Recovery, Concentrate Grade, and Tailings

Grade with Time (Test 7, Table X)

Conditions: Reagent 712, 1.30 lb./ton
pH: 6.7

<table>
<thead>
<tr>
<th>Time of Collection, minutes</th>
<th>Cumulative Recovery %</th>
<th>Grade of Cumulative Grade of 0.185%</th>
<th>Cumulative Concentrate Grade % U₃O₈</th>
<th>Tailings Grade % U₃O₈</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>31.2</td>
<td>0.185</td>
<td>0.185</td>
<td>0.062</td>
</tr>
<tr>
<td>4</td>
<td>46.8</td>
<td>0.177</td>
<td>0.183</td>
<td>0.052</td>
</tr>
<tr>
<td>6</td>
<td>62.2</td>
<td>0.156</td>
<td>0.169</td>
<td>0.041</td>
</tr>
<tr>
<td>8</td>
<td>72.4</td>
<td>0.098</td>
<td>0.153</td>
<td>0.034</td>
</tr>
<tr>
<td>10</td>
<td>79.3</td>
<td>0.047</td>
<td>0.127</td>
<td>0.031</td>
</tr>
<tr>
<td>13</td>
<td>84.2</td>
<td>0.049</td>
<td>0.117</td>
<td>0.028</td>
</tr>
</tbody>
</table>

Feed Analysis = 0.078% U₃O₈

It will be noticed from Fig. 15 that after 8 minutes of collection the recovery does not increase greatly, but there is a serious reduction in the grade of the concentrate. Practically no reduction in the grade of the tailings is accomplished after 8 minutes of collection. If the collection were halted after 8 minutes a concentrate containing approximately 73% of the uranium values at a grade of 0.15% U₃O₈ would be obtained.
VARIATION OF RECOVERY, CONCENTRATE GRADE, AND TAILINGS GRADE WITH TIME USING CYANURED REAGENT 712 (Test 7, Table I)

Fig. 13
Data similar to that obtained for Test 7 were obtained for Test 8 in Table X. The effects of length of collection period on recovery and on the grades of both the concentrate and the tailings are shown in Table XIII and graphically in Fig. 14. Since the recovery is so low, the value of the results presented in Table XIII and Fig. 14 is doubtful. A recovery similar to that obtained in Test 7 would probably not be obtained by extending the collection period, since the froth formed in Test 8 was very weak, probably due to the acidity.

Table XIII - Variation of Recovery, Concentrate Grade, and Tailings with Time (Test 8, Table X)

| Conditions: Cyanamid Reagent 712, 1.30 lb./ton pH, 4.7 |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| Time of Collection (min.) | Cumulative Recovery % | Grade of Cumulative Concentrate Grade (%U₃O₈) | Tailings Grade (%U₃O₈) |
| 2 | 2.0 | 0.285 | 0.285 | 0.069 |
| 4 | 3.3 | 0.319 | 0.298 | 0.070 |
| 6 | 5.5 | 0.296 | 0.298 | 0.068 |
| 8 | 7.8 | 0.308 | 0.300 | 0.069 |
| 10 | 10.2 | 0.289 | 0.298 | 0.068 |
| 13 | 12.3 | 0.281 | 0.292 | 0.068 |

Feed Analysis = 0.074% U₃O₈

The effect of pH on the flotation of uraninite with Reagent 712 may be estimated from Fig. 15. These curves were drawn through the points representing the results of Tests 7, 8 and 9, Table X. The data for Tests 2, 3, 4, 5 and 6 are also plotted on this graph. The fact that the points for Tests 3, 4, 5, and 6 are in relatively close
Fig. 14

VARIATION OF RECOVERY, CONCENTRATE GRADE, AND TAILINGS GRADE WITH TIME USING CYANAMID REAGENT 712 (Test 8, Table X)

Time of Collection (min.)
EFFECT OF pH ON RECOVERY AND CONCENTRATE GRADE
IN FLOTATIONS WITH CYANAMID REAGENT 712

○ Grade - without 1-decanethiol
○ Recovery - without 1-decanethiol
○ Grade - with 1-decanethiol
○ Recovery - with 1-decanethiol
coincidence with the curve drawn through the points representing Tests 7, 8 and 9 leads to the conclusion that neither roasting with sulfur nor the presence of 1-decanthiol has any significant effect on the recovery and on the grade of the concentrate in experiments where Reagent 712 was used. It would appear that a recovery of over 70% of the uranium at approximately twice the grade of the feed can be obtained with Cyanamid Reagent 712 functioning as both collector and frother at a pH of approximately 7.

C. Miscellaneous Flotation Experiments.

This section contains results of flotation experiments which are of some interest but which were not subjected to detailed investigation.

Some tests were carried out in which Sulpho Castor 50+ was the flotation reagent. At the time these tests were conducted the desliming procedure was also being investigated. Hence, the percentage of the total U₃O₈ recovered as slimes was quite large and variable. The conditioning periods in the tests in which Sulpho-Castor 50 was used were necessarily short since the reagent functions as a frother as well as a collector. It should also be noted that in these tests tap water was used. In one test the pulp was conditioned for 4 minutes at 2100 r.p.m. with no pH regulators present. The pH was estimated to be approximately 8. A collection period of 8 minutes, which followed immediately, yielded a concentrate containing 33.6% of the U₃O₈ at a grade of 0.174% U₃O₈. The froth was barren at the end of 8 minutes. The slimes contained 25.3% of the U₃O₈ at a grade of 0.126% U₃O₈. The feed presumably analyzed 0.074% U₃O₈. (The feed to every test was

+ A product of the Dye and Chemical Co. of Canada, Ltd., Kingston, Ontario.
not analyzed at this point in the work). The amount of Sulpho Castor 50 present in the conditioning period was 1.41 lb./ton. During the collection period an additional 0.47 lb./ton was added. The pulp density was approximately 25%.

The effect of pH on floatations of uraninite with Sulpho Castor 50 was briefly investigated. High concentrations of NaOH greatly reduced the volume of the froth. The addition of HCl to neutralize the NaOH greatly increased the froth volume. In one test, in which no NaOH was present, the pH was lowered to 4.1 with HCl. Addition of Sulpho Castor 50 raised the pH to 5.6. The pulp was conditioned for 3 minutes at 2100 r.p.m. with 0.4 lb. of Sulpho Castor 50/ton. During the 8 minute collection period which followed immediately, an additional 0.60 lb./ton were added. The froth was barren at the end of 8 minutes collection. The concentrate contained 18.7% of the U₃O₈ at a grade of 0.183% U₃O₈. The slime fraction contained 37.0% of the U₃O₈ at a grade of 0.115% U₃O₈. The pulp density was 22% and the feed was estimated to be 0.074% U₃O₈.

The best result obtained in the Sulpho Castor 50 work was a recovery of 79.1% of the U₃O₈ in a fraction analyzing 0.127% U₃O₈. (The feed analysis was approximately 0.074% U₃O₈). This material was a combination of the slimes, the concentrate from a "rougther" flotation, and the concentrate from a "scavenger" flotation. In the rougher flotation the pulp was conditioned with 0.52 lb. of Sulpho Castor 50 for 3 minutes at 2100 r.p.m. During the 9 minute collection period an additional 0.17 lb./ton of the collector-frother was added. The froth was barren after 9 minutes. The pH was 7.9 and the pulp density was 25.0% during this flotation. The slimes contained 44.8% of the U₃O₈ and the rougher concentrate contained 24.0% of the U₃O₈. The grade of the slimes was 0.14% U₃O₈ and the grade of the concentrate was 0.12% U₃O₈.
In the scavenger flotation the conditioning period with 0.5 lb. of Sulpho Castor 50 per ton was 5 minutes. An additional 0.5 lb./ton was added during the 8 minute collection period. The froth was barren after 8 minutes. The pH and the pulp density were not measured. The scavenger concentrate contained 10.3% of the original $\text{U}_3\text{O}_8$ at a grade of 0.10% $\text{U}_3\text{O}_8$. While a recovery of almost 90% of the $\text{U}_3\text{O}_8$ is encouraging, it must be remembered that in this test over one half the total recovery was obtained in the slime fraction. The actual amount recovered by flotation was only 34.3% of the original $\text{U}_3\text{O}_8$.

Tributyl phosphate was briefly investigated as a collector for uraninite in Charlebois Lake uranium ores. At a pH of 11.5 (sodium hydroxide) 20 minute conditioning with 0.15 lb. of tributyl phosphate per ton of ore yielded a concentrate containing 24.2% of the $\text{U}_3\text{O}_8$ at a grade of 0.18% $\text{U}_3\text{O}_8$ from a feed analyzing 0.072% $\text{U}_3\text{O}_8$. The pulp density was 26.0% dry solids by weight. The slime fraction contained only 3.7% of the $\text{U}_3\text{O}_8$ at a grade of 0.22% $\text{U}_3\text{O}_8$. The frother was Dowfroth 250 : 0.24 lb./ton. The froth was collected for 5 minutes, at the end of which time it was barren. As this result is no better than those obtained by the use of 1-decanethiol as collector under similar conditions (Table VIII) experiments with tributyl phosphate were abandoned.

The use of sodium sulfide as an activator for uraninite was briefly mentioned in connection with the work done on 1-decanethiol and Dowfroth 250 as flotation reagents. Activation by sodium sulfide was further investigated in floatations in which a typical sulfide collector, sodium xanthate, was used. One test was conducted on material ground for 20 minutes and another on material ground for 45 minutes in
the ball mill under the conditions previously specified. In the test

carried out on the material ground for 20 minutes, the amounts of re-
agents used were: sodium sulfide, 0.48 lb./ton; sodium xanthate, 1.0 lb./ton; and Dowfroth 250, 0.24 lb./ton. The "preconditioning" and "conditioning" periods were each of 10 minutes duration. In 5 minutes collection a concentrate containing 41.2% of the uranium values at a grade of 0.18% U₃O₈ was obtained from a feed analyzing 0.075% U₃O₈. The froth was barren at the end of the collection period. The pH and pulp density for this flotation were 8.7 and 24.0%, respectively. The slimes contained 6.5% of the U₃O₈ at a grade of 0.22% U₃O₈. The material ground for 45 minutes was tested similarly with the exception that the amount of Dowfroth 250 was 0.38 lb./ton and the "preconditioning" and "conditioning" periods were 20 minutes and 5 minutes, respectively. The pH was 7.5 and the pulp density was approximately 25%. In this case the concentrate contained 35.0% of the U₃O₈ at a grade of 0.12% U₃O₈. The froth was not barren at the end of the 5 minute collection period. The feed to this test analyzed 0.074% U₃O₈. The slimes contained 20.2% of the U₃O₈ at a grade of 0.12% U₃O₈. It is thought that this decrease in grade was the result of overgrinding rather than the result of the increased amount of frother. While it is not likely that the additional size reduction produced a surface area too large for the amount of reagent present, it is possible that the reduction in the grade of the concentrate is due to contamination by slime gangue. The fact that the ratio of gangue minerals to uraninite was higher in the finer fractions of the second experiment is evidenced by the decreased grade of the slime fraction.

Although the investigations of Sulpho Castor 50, tributyl
phosphate, and sodium xanthate as collectors for uraninite were brief, they do reveal some interesting lines of experimentation which could be followed. The results obtained with sodium xanthate as collector and sodium sulfide as activator are particularly encouraging. Further investigations of these conditions with increased amounts of frother would be interesting in view of the improved results obtained by using additional frother with 1-decanethiol.

V. Conclusions.

This investigation has been concerned with the concentration of uraninite by flotation from a typical low-grade uranium ore of the pegmatite type from the Charlebois Lake area of north-eastern Saskatchewan. The majority of this flotation work was carried out with a 500 gram Denver Sub-A flotation cell. The following conclusions may be drawn from the results of the investigations reported in this work.

(1) The 50 gram flotation cells were found to be unsuitable for quantitative work, at least when operating as agitation cells without sub-aeration.

(2) In the experiments in which various thioalcohols were used as collectors, the number of carbon atoms in the mercaptan seemed to have no significant effect on either the grade of the concentrate or the recovery at a pH of 11.5.

(3) When using the thioalcohols as collectors and Dowfroth 250 as frother, it appears necessary to have an alkaline medium to obtain a stable froth. In the experiments in which the collector was 1-decanethiol (0.89 lb./ton) and the frother was Dowfroth 250 (0.24 lb./ton) the maximum recovery was obtained at a pH of 10.7.

(4) The results of the screen and radiometric analyses conducted on the
accumulated tailings indicate that in flotation where there are high losses of $U_3O_8$ in the tailings, the $U_3O_8$ remaining in the cell after flotation is in the fines. It would seem that 20 minute grinding in the ball mill under the conditions specified is sufficient to liberate the majority of the uraninite.

(5) The results of contact angle measurements indicate that Cyanamid Reagent 712 shows some selectivity for uraninite in the flotation operation. Quartz was the only gangue mineral showing a finite contact angle after treatment with a 1% solution of Reagent 712. Thus, it would seem that quartz would be the only gangue mineral to interfere in the flotation of uraninite with Reagent 712. The relatively low grade of the concentrates obtained in the flotations of uraninite with Reagent 712 indicates that the concentrates are being contaminated by gangue. This contamination is probably partly due to the fact that gangue particles were mechanically carried over in the froth. The grade of the concentrate might be increased by reducing the concentration of Reagent 712 to obtain a less vigorous froth.

(6) Recoveries of approximately 80% of the $U_3O_8$ can be obtained by using Cyanamid Reagent 712 and adding this reagent intermittently throughout the collection period. The rate of addition of Reagent 712 should be governed by the character of the froth. The grade of the concentrate obtained was approximately twice that of the feed. In flotations with Cyanamid Reagent 712 the maximum recovery was obtained at a pH of approximately 7. The optimum time of collection was approximately 8 minutes. Thus, the conclusion, drawn from contact angle measurements, that Cyanamid Reagent 712 showed selectivity towards uraninite was corroborated by the results of flotation experiments.
Other conclusions were drawn at various points throughout the course of this work. Since they apply only to the particular experiments for which they were mentioned, they will not be restated here.

VI. Suggestions for Future Work.

Some of the preliminary investigations conducted in the present work indicate some interesting lines of investigation which could be followed.

(1) A complete investigation of the use of Cyanamid reagent 712 as a flotation reagent should be carried out. This investigation should include studies on the effects of depressors, activators, pH, temperature, pulp density, degree of grind, impellor speeds, and rate of addition of air and reagent on the recovery and on the grade of the concentrate.

(2) The studies initiated on the flotation of uraninite with sodium sulfide as activator and sodium xanthate as collector could be continued.

With regard to the procedure used for carrying out the flotation tests the author would like to suggest the following:

(1) Possibly a scrubbing step should be added to the flotation procedure. This would be done immediately prior to the desliming step. It would involve thoroughly agitating the ore at a pulp density of 40% to 60% solids by weight. Sulfuric acid, sodium hydroxide or sodium silicate could be used as cleansing agents. This modification in the procedure would be of assistance in removing slime coating.

(2) The practice of obtaining the concentrate in fractions based on the time of collection should be employed more often than it has been in the past. A great deal of information is obtainable from graphs of the type shown in Fig. 13 and Fig. 14.
LIST OF REFERENCES.

8. Queen's University Progress Reports supplied by the Mines Branch, Ottawa.
15. Perkins. U. S. Patent 1,564,504 (1921), as reported in Ref. 14, p.5.


APPENDIX A.

EFFECTS OF WEAR IN THE BALL MILL.

At the conclusion of the experimental work reported in this thesis an investigation was made in an attempt to determine if wear in the ball mill was affecting the size reduction being given the ore. At the conclusion of this work the ball mill had been run approximately 42 hours from the time it was put into operation in June of 1955. At the end of this time the balls were removed from the mill and reweighed. The 1.5 in. nominal diameter balls had been reduced in weight from 5 lb. to 4.8 lb. The 1 in. nominal diameter balls had been reduced in weight from 5 lb. to 4.8 lb. The 0.75 in. nominal diameter balls had been reduced in weight from 5 lb. to 4.3 lb. The net weight of the ball load remaining in the mill after 42 hours of operation was 15.9 lb. The original weight of balls was 15.0 lbs.

In order to determine more accurately the loss in weight of the balls, the following procedure was adopted. Samples of 5 of the 1.5 in., 1 in. and 0.75 in. nominal diameter balls removed from the mill were taken. Similar samples were taken from a group of balls which had not been used during the course of this work. For the "used" balls the mean weights were: 1.5 in. nominal diameter, 221 grams; 1 in. nominal diameter, 67.5 grams; 0.75 in. nominal diameter, 50.6 grams. For the "unused" balls the mean weights were: 1.5 in. nominal diameter, 226.7 grams; 1 in. nominal diameter, 70.4 grams; 0.75 in. nominal diameter, 31.8 grams. The actual data are shown in Table XIV. While the mean weights of the balls before and after use were not treated statistically to test the significance of the differences in the means, the data presented in Table XIV seem to indicate definitely that appreciable wear is occurring in the ball mill.
Table XIV. Comparative Weights of "Used" and "Unused" Balls.

Weights of "Used" Balls (grams).

<table>
<thead>
<tr>
<th>1.5 in. nominal diam.</th>
<th>1 in. nominal diam.</th>
<th>0.75 in. nominal diam.</th>
</tr>
</thead>
<tbody>
<tr>
<td>220.7</td>
<td>66.7</td>
<td>30.0</td>
</tr>
<tr>
<td>225.2</td>
<td>67.8</td>
<td>30.5</td>
</tr>
<tr>
<td>219.7</td>
<td>67.7</td>
<td>30.9</td>
</tr>
<tr>
<td>220.9</td>
<td>66.9</td>
<td>30.7</td>
</tr>
<tr>
<td>220.4</td>
<td>68.5</td>
<td>30.7</td>
</tr>
<tr>
<td>Mean = 221.0</td>
<td>Mean = 67.5</td>
<td>Mean = 30.6</td>
</tr>
</tbody>
</table>

Weights of "Unused" Balls (grams)

<table>
<thead>
<tr>
<th>1.5 in. nominal diam.</th>
<th>1 in. nominal diam.</th>
<th>0.75 in. nominal diam.</th>
</tr>
</thead>
<tbody>
<tr>
<td>226.9</td>
<td>69.2</td>
<td>31.2</td>
</tr>
<tr>
<td>225.0</td>
<td>70.4</td>
<td>31.5</td>
</tr>
<tr>
<td>227.7</td>
<td>70.3</td>
<td>32.3</td>
</tr>
<tr>
<td>227.4</td>
<td>70.8</td>
<td>32.3</td>
</tr>
<tr>
<td>226.6</td>
<td>71.4</td>
<td>31.5</td>
</tr>
<tr>
<td>Mean = 226.7</td>
<td>Mean = 70.4</td>
<td>Mean = 31.8</td>
</tr>
</tbody>
</table>

To test the effect of ball mill wear on the size reduction operation a screen analysis of material ground for 20 minutes in the ball mill under the usual conditions was conducted. This grinding was done with the "used" balls. The data are presented in Table XV. These may be compared with similar data for the 20 minute grind presented in Fig. 4 and Table II. The data in this figure and table applied at the end of approximately one hour of operation of the ball mill. It will be noticed that the greatest difference in the screen analyses is in the -400 mesh fraction; otherwise the screen analyses are in relatively good
agreement. It is thought that the changes in the screen analysis which took place during the course of the work would not significantly affect the results of the flotation experiments.

Table XV. Screen Analysis of Ore DE 55-1 Ground in Ball Mill for Twenty Minutes.*

<table>
<thead>
<tr>
<th>Tyler Standard Screen Mesh</th>
<th>Weight % Retained</th>
<th>Weight % Passing Screen</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>Trace</td>
<td>100.0</td>
</tr>
<tr>
<td>10</td>
<td>0.2</td>
<td>99.8</td>
</tr>
<tr>
<td>14</td>
<td>0.3</td>
<td>99.5</td>
</tr>
<tr>
<td>20</td>
<td>0.2</td>
<td>99.3</td>
</tr>
<tr>
<td>28</td>
<td>0.3</td>
<td>99.0</td>
</tr>
<tr>
<td>35</td>
<td>0.2</td>
<td>98.8</td>
</tr>
<tr>
<td>48</td>
<td>0.2</td>
<td>98.6</td>
</tr>
<tr>
<td>65</td>
<td>3.1</td>
<td>95.5</td>
</tr>
<tr>
<td>100</td>
<td>8.8</td>
<td>86.7</td>
</tr>
<tr>
<td>150</td>
<td>12.8</td>
<td>73.9</td>
</tr>
<tr>
<td>200</td>
<td>15.9</td>
<td>58.0</td>
</tr>
<tr>
<td>270</td>
<td>13.6</td>
<td>44.0</td>
</tr>
<tr>
<td>400</td>
<td>20.9</td>
<td>23.5</td>
</tr>
<tr>
<td>-400</td>
<td>25.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>100.0</td>
<td></td>
</tr>
</tbody>
</table>

+ Based on sum of weights of fractions: 216.8 grams. Weight of feed was 216.0 grams.

* When this grinding was done, the balls had been in use for approximately 42 hours. These data should be compared with those given in Table II. The grinding conditions used to obtain the data in Table II were similar to those used to obtain the data in Table II with the exception that in the former case the balls had been in service for approximately one hour.
APPENDIX B.

DETAILS OF PROCEDURE FOR MEASURING CONTACT ANGLES.

An excellent diagram of the apparatus used by the author for the measurement of contact angles is given by Talbot (1). In the measurement of contact angles in the present work the following procedure was adopted. The mineral specimens were polished with 4/0 emery paper until distilled water did not draw back from the surface when the sample was rinsed. The specimen was then placed under distilled water in the glass cell of the "bubble machine." An attempt was made to attach an air bubble to the surface of the solid. The air bubbles were formed by adjusting the screw of a micro-pipettor attached to a piece of capillary tubing approximately 4 in. long. The capillary tubing and pipettor were suspended above the apparatus. When the specimen was in position in the cell the capillary tube and pipettor were lowered until the tip of the capillary was approximately 0.06 in. above the surface of the solid. An air bubble was brought into contact with the surface by adjusting the screw of the micro-pipettor. The bubble was forced against the surface of the specimen and left in that position for approximately 5 minutes. If, after that time, the bubble would not adhere to the specimen when the capillary tube was raised, the surface was assumed to be relatively free of contamination. It should be emphasized that the bubble was blown by the micro-pipettor and was attached to the capillary tube throughout the preceding operation. If the bubble showed any tendency to adhere, the surface of the specimen was repolished and the foregoing procedure was repeated. If the bubble showed no tendency to adhere to the surface the specimen was removed from the glass cell and placed in the conditioning medium for the desired length of time. Upon removal from the conditioning medium the sample was thoroughly rinsed in a stream of distilled
water and replaced in the glass cell of the "bubble machine". A bubble was brought to the surface of the specimen as described previously. If the bubble would not adhere when the capillary tube was raised slightly, the contact angle was recorded as zero. Occasionally a bubble was noted which showed some tendency to adhere when the capillary tube was so raised. In such cases the bubble elongated as the capillary was withdrawn from the surface, but the bubble was not stable at the surface without being held down by the capillary tube. Such cases were recorded as exhibiting a zero angle of contact.

A finite contact angle was taken as the case where the bubble adhered to the surface of the specimen without being held down by the capillary tube. In such instances the capillary tube was completely removed from the apparatus. The image of a bubble which adhered to the specimen without being held down by the capillary was magnified through a microscope. The optical system consisted of a 2.5X ocular lens and a 52 mm. focal length, 0.10 N.A. + objective lens. The image of the bubble was focused on a ground glass screen and was traced onto a piece of white paper. The angle of contact was then measured directly by means of a protractor.

+ N.A. means "numerical aperture", a measure of the resolving power of the lens.
The data shown in Table XVI were accumulated as a check on the reproducibility of the experiments carried out with the 500 gram Denver Sub-A flotation cell. The conditions for the experiments have been stated in the section on the development of the procedure. Conditions not mentioned there were as follows:

**Feed:** Sample DE 55-1

**Flotation Reagent:** Cyanamid Reagent 712, 1.3 lb./ton.

**pH:** 6.8 to 7.4.

**Pulp Density:** 26% to 30%

**Collection Time:** 15 minutes. At the end of the collection period the froth was barren.

Comparison of the results presented in Table XVI with those contained in Tables V and VI leads to the conclusion that the experiments performed with the 500 gram cell are considerably more reproducible than those performed with the 50 gram flotation cells.

It will be noticed that the recovery in the concentrate of the fourth test reported in Table VI is considerably lower than the corresponding recoveries in the other tests reported in this table. In all tests other than the fourth the medicine dropper was held horizontally while delivering reagent. In the fourth test the pipette was held at an angle of approximately 45°. It has since been found that at an angle of approximately 45° the dropper delivers approximately 32% less reagent than when the dropper is held horizontally. It is interesting to note that the recovery in the concentrate of the fourth test in Table XVI is approximately 34% lower than the mean of the corresponding recoveries in the other six tests reported in this table.
Table XVI - Results of Replicate Experiments Conducted with the 500-Gram Denver Sub-A Flotation Cell.

<table>
<thead>
<tr>
<th>Feed Analysis</th>
<th>Slimes</th>
<th>Concentrate</th>
<th>Tailings</th>
</tr>
</thead>
<tbody>
<tr>
<td>% U₃O₈</td>
<td>% U₃O₈</td>
<td>Grade</td>
<td>% U₃O₈</td>
</tr>
<tr>
<td>0.077</td>
<td>0.156</td>
<td>0.6</td>
<td>0.109</td>
</tr>
<tr>
<td>0.078</td>
<td>0.177</td>
<td>0.6</td>
<td>0.117</td>
</tr>
<tr>
<td>0.080</td>
<td>0.169</td>
<td>2.8</td>
<td>0.120</td>
</tr>
<tr>
<td>0.078*</td>
<td>0.175</td>
<td>6.8</td>
<td>0.140</td>
</tr>
<tr>
<td>0.074*</td>
<td>0.180</td>
<td>9.1</td>
<td>0.100</td>
</tr>
<tr>
<td>0.074*</td>
<td>0.188</td>
<td>8.7</td>
<td>0.117</td>
</tr>
<tr>
<td>0.079*</td>
<td>0.206</td>
<td>9.6</td>
<td>0.127</td>
</tr>
</tbody>
</table>

+ The concentration of Reagent 712 was low in this test.

* The author is indebted to Mr. P. D. Manwaring for these results.