MAY 0 1974

STATEMENT OF ROBERT A. RAGOTZKIE

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MADISON, WISCONSIN

MONS 204513

Qualifications and Experience

My name is Robert A. Ragotzkie and I am Professor of Meteorology and Environmental Studies and Director of the Marine Studies Center, University of Wisconsin-Madison. I hold a B.S. in biology (1948) and M.S. in sanitation (1950) from Rutgers University and a Ph.D. in meteorology-zoology (1953) from the University of Wisconsin. I have done research on inland lakes, the Great Lakes and estuaries, specifically on currents and circulations of these water bodies and on the inter-relationships of water movements and biological populations. I have published numerous papers on these subjects (see attached list).

Statement

I have been asked to comment on the EPA Proposed Toxic Effluent Standards as published in the Federal Register, December 27, 1973, vol. 38, number 247, Part II, pp. 35388-35395 and on the statements and affidavits on these proposed standards by various interested parties. My comments are limited to the hydrodynamic aspects of the proposed standards.

Hydrodynamic Model for Lakes

As pointed out in the affidavits of Carlos M. Fetterolf, Jr. for the State of Michigan, Albert C. Clark for the Manufacturing Chemists Association, John L. Mancini for the American Cyanamid Company and others the definition and hydrodynamic specifications applied to lakes are oversimplified and therefore to some degree necessarily arbitrary.

The following discussion will demonstrate that it is not necessary to use the 7 day once in 10 year low flow calculation to derive the chronic protection part of the effluent standards. It should be noted that for lakes the use of the mean annual outflow is the correct and more appropriate flow to be used.

The definition of a lake in terms of retention time or hydrologic flushing time is inevitably arbitrary. However, whenever the flow of a stream or river is interrupted and the water retained for a discrete period of time the pollutant loading characteristics of the body of water

are changed.

To illustrate this, consider the discharge of a waste into a flowing stream. Disregarding the concept of mixing zone for the moment, the waste loading of the stream is simply calculated as the pollutant concentration of the waste stream times its flow rate (mass emission) divided by the flow rate of the receving stream or river. In the case of the discharge of a waste into a lake however, the concentration of the pollutant builds up gradually to an ultimate limit which is theoretically equal to that of a stream with a flow equal to the outflow of the lake in question. The time of this, as described below, depends on the hydrologic flushing time of the lake or impoundment, reaching 95% of the ultimate concentration in 3 times the flushing time (Raney 1967).

$$T = V/Q$$

where T = flushing time

V = volume of lake

Q = annual mean flow of outlet

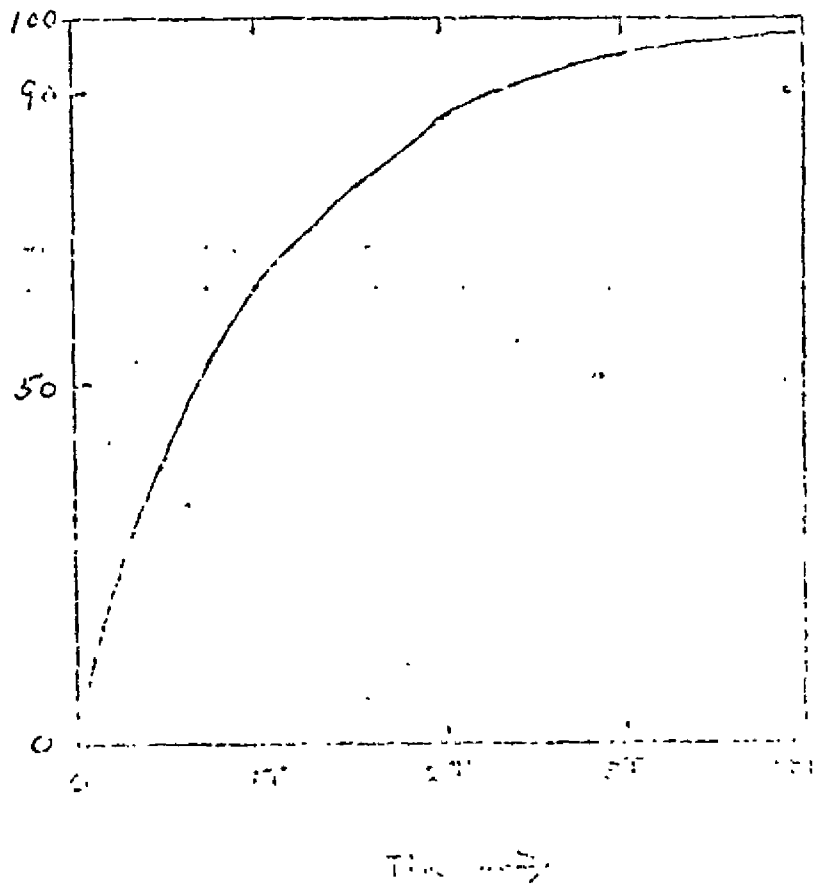
and $$C_L = C_{IN}(1 - e^{-t/T})$$

where C_L = concentration in lake

C_{IN} = concentration in inflow

t = time

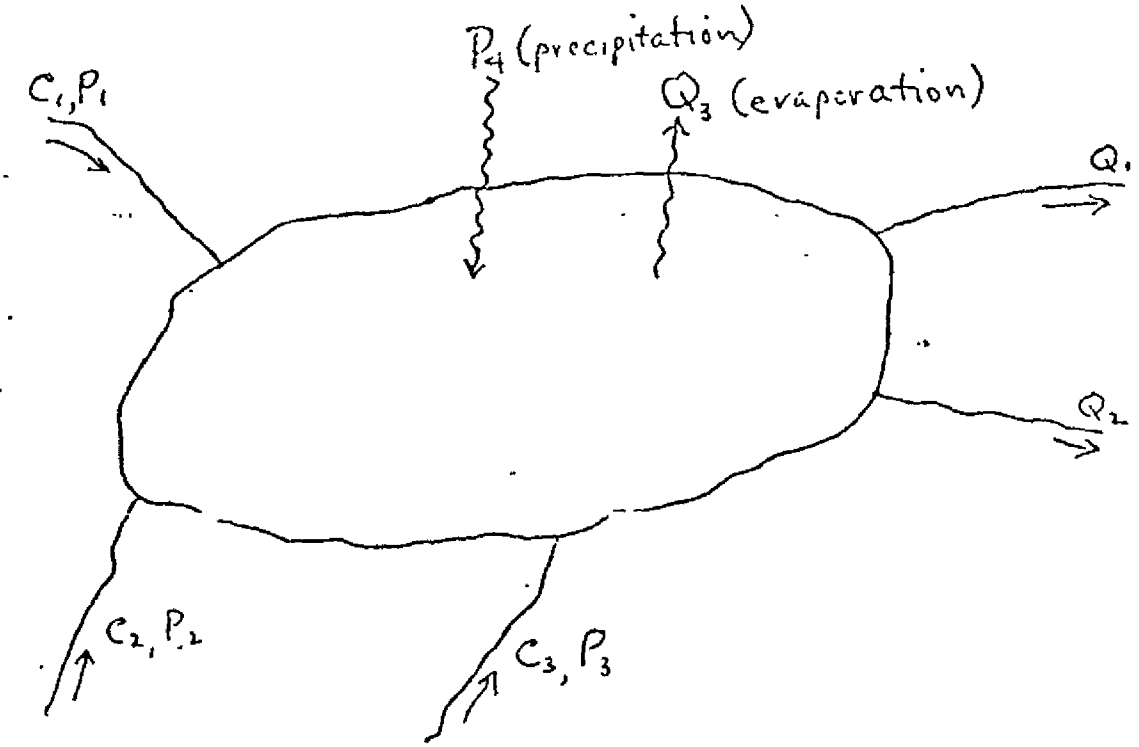
The rate of buildup of a pollutant in a lake is shown graphically below.



PERCENT OF EQUILIBRIUM POLLUTANT CONCENTRATION

Time (yr)

This simple lake model can be generalized to the case where there is more than one inflow to and outflow from the Lake, and to include precipitation (a nonpolluted inflow) and evaporation (a nonpolluted outflow). Note that it is not necessary to assume precipitation equal to evaporation.



We then get for the concentration in the lake

$$C_L = \frac{\Sigma C_i P_i}{\Sigma Q_j} \left(1 - e^{-t/T} \right)$$

where $\Sigma C_i P_i = C_1 P_1 + C_2 P_2 + C_3 P_3$ for the case shown

$\Sigma Q_j = Q_1 + Q_2$ for the case shown

P_i = flow rate into the lake from river (i)

Q_i = flow rate out through river (j)

C_i = pollutant concentration in river (i)

Thus, the ultimate steady state concentration level for a pollutant is $\Sigma C_i P_i / \Sigma Q_i$. For a more detailed discussion of this model approach see Rainey (1967).

This model is based on the following assumptions:

1. The substance in question is conservative.
2. The lake undergoes complete mixing in a time small compared to T.

The conservative assumption means that the concentration in water of the variable (toxic substance) in question is changed only by avection and diffusion processes, i.e. by steady flow or by mixing with water of different concentration. Changes in concentration owing to biochemical degradation or transformation or to permanent sedimentation can be taken into account as a loss factor modification of the concentration model for the body of water in question.

It should be pointed out, however, that loss by permanent sedimentation without biochemical degradation, e.g. as in the case of heavy metals, may be detrimental to the aquatic system in that concentration in the sediments can have a deleterious effect on benthic organisms and their predators. Therefore this type of non-conservative behavior of toxic substances in the aquatic system may require downward adjustment of effluent standards and loading in order to protect the benthic environment.

On the other hand, biochemical degradation of toxic substances may effectively reduce their concentration and transform them to less harmful or harmless substances. In this case consideration can be given to the relaxation of effluent standards, based on the rates of degradation in the natural environment.

In summary then, non-conservative behavior of pollutants may result in the necessity to adjust effluent standards and loading in either direction depending on the nature and final result of the process.

The complete mixing assumption is probably met when the time scale is a year or more except in the cases of meromictic lakes or the Great Lakes. For shorter time scales the problem of a suitable mixing zone arises.

Recognizing the mixing zone problem as separate, the concept of a lake or impoundment as simply a part of a stream where the gravity flow is interrupted is the simplest and most useful model to use to establish chronic toxic loading. Furthermore since a lake effectively integrates the flow of the stream, the annual average inflow and outflow are the proper statistics to use in computing toxic loading. The 7-day low flow criterion is meaningless because this is averaged out by the storage capacity of the lake. In the case of lakes or impoundments with a flushing time less than 1/3 of a year (4 months) the low flow for a period equal to 3 times the flushing time could be substituted for the mean annual outflow.

Therefore with the exception of the special cases listed below and the Great Lakes, it is recommended that chronic toxic loading for lakes be based on the mean annual outflow or low flow for 3 times the flushing time for lakes with a flushing time less than 4 months.

Special Cases for Lakes

1. Meromictic lakes

These natural lakes fail to mix vertically each year because of permanent density stratification due to the concentration of dissolved materials in the lower layer. Most of these lakes are small and have low inflow-outflow rates. It is recommended that discharge of toxic substances into meromictic lakes be prohibited.

2. Great Salt Lake

The affidavit of Donald B. Porcella for Kennecott Copper Corporation on the case of Great Salt Lake, Utah clearly points out the need for special treatment for the control of toxic effluents into this body of water. Because of its high evaporation and lack of outlet Great Salt Lake has a very high salt content. It contains a relatively simple ecosystem and the effects of any toxic discharge to the lake should be measured against the endemic organisms in this ecosystem and any possible hazard to human health.

3. Seepage lakes

These include lakes fed by groundwater or bogs and which have either no surface outflow or intermittent surface outflow. These are mostly small lakes in wilderness or recreation areas. It is recommended that discharge of toxic substances into seepage lakes be prohibited.

Lower Size Limit for Lakes

Albert Clark's affidavit for the Manufacturing Chemists Association and Carlos M. Fetterolf for the State of Michigan question the 500 acre

minimum size for lakes which should receive toxic effluents. Fetterolf correctly points out that volume is a better statistic than area. Clark questions the scientific validity of any minimum size.

Except in consideration of ordinary aesthetic sensitivity there is no clearcut biological or ecological basis for setting a lower size threshhold for lakes below which toxic discharge would be prohibited. The mixing zone problem may be severe in certain very shallow lakes however, where chronic toxic loading could easily be exceeded due to the inhibition of horizontal mixing by extensive rooted aquatic vegetation. Permissible toxic loading should be determined on the basis of mean surface outflow. Very small lakes may have quite short flushing times, especially on chains of lakes or flowages. As recommended in the "Hydrodynamic Model" section, for lakes with flushing times shorter than 1/3 year chronic toxic loading should be based on low flow for a period 3 times the flushing time.

Lower Size Limit for Streams

The Manufacturing Chemists Association and other industries object to the prohibition of any discharge into streams with a low flow less than 10 cfs. There is no scientific justification for selecting 10 cfs as a cutoff point. It would seem that effluent standards based on loading of the receiving water that will protect larger streams will also protect smaller ones.

It is recommended that this low flow cutoff be deleted from the regulations.

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The Great Lakes

In his affidavit for the State of Michigan Carlos M. Fetterolf, Jr. properly calls attention to the special characteristics of the Great Lakes and their similarities to coastal waters of the sea.

The Great Lakes have characteristics of both lakes and oceans. They are extremely large bodies of fresh water, receive water from land drainage and, except for Lake Superior, from the outflow of the next upstream lake. The outflows are very large ranging from 75,000 cfs from Lake Superior to 240,000 cfs from Lake Ontario. Their flushing times vary from about 183 years for Lake Superior to 2.7 years for Lake Erie (see Table 1).

All of the Great Lakes undergo thermal stratification in summer and weak thermal stratification in winter. All five mix vertically in spring and fall. The circulation of the Great Lakes is primarily wind-driven and is characterized by coastal currents which in some locations and at some times are jet-like in character. These coastal currents may be persistent in direction for several months (e.g. the Keweenaw Current in Lake Superior) or they may undergo reversal every few days (e.g. those observed in Lake Huron and Lake Michigan). These currents may extend 1 to 10 miles offshore and often exhibit a fine structure which suggests jet-like currents imbedded in the overall current system.

The Great Lakes, being closed basins, are also subject to internal oscillations and other wind-driven motions leading to episodes of upwelling. These vertical movements result in deeper, hypolimnion water rising to the surface along the coastline. The upwelling episodes are usually of a few days duration or less and are followed by a return of the hypolimnion water to deeper levels.

Since all waste effluents enter the lakes at the shoreline or nearly so, the movements and dispersal of the waste are governed by the speed, direction, lateral extent and persistence of the coastal currents and by the frequency and

TABLE 1

Hydrologic Statistics for the Great Lakes

Lake	Area (ft^2) (km^2)	Mean Depth (ft) (m)	Volume (ft^3) (km^3)	Outflow (cfs) (m^3/sec)	Flushing Time (yrs)
Superior	834×10^9	489	4.32×10^{14}	74,580	183
	82,103	149	12,234	2,112	
Michigan	622×10^9	279	1.73×10^{14}	48,040	114
	57,757	85	4918	1360	
Huron	841×10^9	195	1.25×10^{14}	179,080	22
	59,570	59.4	3539	5070	
Erie	276×10^9	62	0.171×10^{14}	204,140	2.7
	25,667	18.9	483.5	5781	
Ontario	205×10^9	283	0.579×10^{14}	240,425	7.6
	19,010	86.3	1638	6808	

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extent of upwelling at the location in question. Thus the coastal hydrographic behavior of the Great Lakes is analogous to coastal waters of the oceans with respect to coastal currents, and in addition is subject to large-scale vertical motions (upwelling) immediately adjacent to the coast, a phenomenon which is less common along the U.S. ocean coasts.

For the purpose of controlling toxic waste discharges the Great Lakes coastal zone can be divided into two general physical environments: (1) open coasts and (2) embayments.

1. Open Coasts

For the open coast the low flow discharge approach for coastal waters of the ocean proposed by Dr. Hugo B. Fischer in his affidavit is appropriate. Fischer suggests that conservative values for coastal currents and mixing rates be adopted and the coastal flow available for dilution calculated therefrom.

Since thermal stratification of the Great Lakes is an important factor affecting vertical mixing, Fischer's suggested values should be modified to take this into account. The suggested modifications are as follows:

Value	Ocean Coast (Fischer)	Great Lakes Coast
minimum current available for dilution	0.1 ft/sec	0.1 ft/sec
minimum vertical spread of the effluent plume	1/4 depth	1/4 depth of summer thermocline or 1/4 depth to bottom, whichever is less
minimum horizontal spread of the effluent plume	10 depths	10 x depth of summer thermocline or 10 x depth to bottom, whichever is less

The effective dilution flow is given by

$$Q = (0.1) \times (0.25d) \times (10d) = 0.25d^2$$

where d is the depth in feet of the summer thermocline or depth to the bottom, whichever is less, and Q is flow in cfs.

The values for the Great Lakes coast are conservative. In some situations the flow of water available for dilution will exceed the flow derived from these values. As in the ocean coast case exceptions could be made if the discharger can show that a larger flow available for dilution consistently exists. Such a demonstration will require monitoring of currents, probably for an entire year, in order to establish the minimum coastal flow in the vicinity of the discharge. Dispersion devices which increase the mixing rate beyond the values based on the depth of the thermocline or to the bottom may also increase the effective available flow.

2. Embayments

The Great Lakes have numerous embayments with a limited connection to the lake as a whole. Examples are Chequamegon Bay and Thunder Bay in Lake Superior, Green Bay and Grand Traverse Bay in Lake Michigan, North Channel, Georgian Bay and Saginaw Bay in Lake Huron. These bays behave more like estuaries in that they are mixing basins where rivers of land drainage water become gradually mixed with and diluted by lake water. This mixing process is driven primarily by surface and internal seiche movements with wind mixing playing a part in some situations. At the mouth of the bay the bay water interacts with the lake as a whole by a pulsing action whereby the bay water moves in and out in response to seiche activity (analogous to tides in the ocean)

and is then entrained in the coastal current moving past the bay opening.

Because of the nature of this circulation in bays, waste loading should be determined by the method applied to estuaries using the tidal prism approach. Each bay would require separate consideration. However, several of the bays have already been studied and flushing models are available.

Chequamegon Bay in Lake Superior at Ashland, Wisconsin, a semi-enclosed bay without a sill, exchanges water with Lake Superior by means of seiche oscillations (Ragotzkie et al, 1969). The surface seiche which displays several modes has a vertical range of 10 cm. or greater 85 percent of the time. Considering only the 140 minute seiche for which the node is at or near the mouth of the bay, a 10 cm. vertical oscillation results in a flushing rate of the bay of 5 percent per day. This "flushed" water is transported away from the mouth of the bay by coastal currents of the lake itself. If the reasonable assumption is made that none of the "flushed" water returns to the bay, this flushing rate per day is exactly the same as the "tidal exchange rates" term employed by Fischer in his affidavit to calculate the effective flow available for dilution.

To complete the tidal estuary analogy and adopting Fischer's method the terms in his equation (1) can be redefined as follows:

$M = (Q + Q_c + RP/T)C$ (from Fischer)

M = discharge rate of material (same)

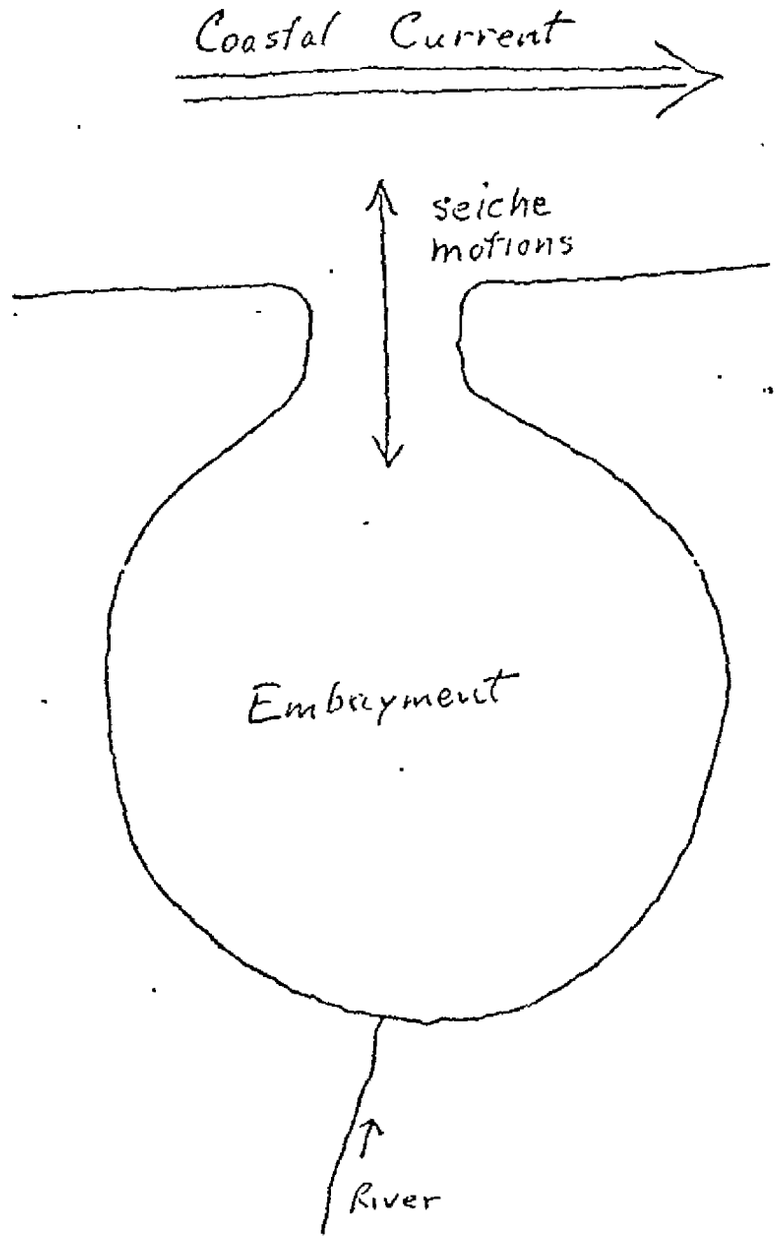
Q = tributary discharge (same)

Q_c = waste stream discharge (same)

R = tidal exchange ratio (seiche flushing rate)

P = tidal prism upstream from discharge point (volume of bay landward from point of discharge

GREAT LAKE EMBAYMENT CIRCULATION



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T = duration of tidal cycle (period of seiche)

C = concentration of material discharged (from Fischer)

Green Bay, Lake Michigan is a larger bay with a multiple opening to Lake Michigan. Its water quality is influenced primarily by the discharge of the Fox River although several other rivers and their associated pollution loading make minor contributions to the system. In this bay effective horizontal diffusion along the long axis is the primary process transporting pollutants from the head to the mouth of the bay. Based on the gradient of the Fox River water from the head to the mouth of the bay the effective diffusion or movement of Fox River water has been determined (Ahrnsbrak and Ragotzkie, 1970). The diffusion coefficient and its variation along the long axis of the bay is largely explained by the motions of the surface seiche.

Based on the diffusion measurements a flushing model for the bay itself has been developed and adapted to computer solution (Ahrnsbrak, 1971) . By this model the rate of buildup or flushing of a pollutant with a source in the bay as well as the concentration distribution can be readily calculated and displayed.

It is therefore recommended that embayments of the Great Lakes be treated in the same manner as ocean estuaries. Each bay will require investigation in order to determine its flushing rate with the lake as a whole and in the larger bays diffusion rates will need to be estimated from measurements of gradients of river water inflow.

All of the above discussion has dealt with the Great Lakes as if they were oceans with estuaries feeding into them. However, there is a fundamental difference between the Great Lakes and the ocean. The ocean is normally

considered a sink for waste with its immense volume being adequate to absorb and disperse waste materials to very low and non-deleterious concentrations. Even this concept is being challenged for certain types of pollutants such as oil which do not readily disperse throughout the entire ocean water volume. The Great Lakes however are not considered as permanent sinks for wastes, especially toxic substances. The lakes form a chain with very large flow rates, especially in the lower lakes, Erie and Ontario. Their flushing times are variable (Table 1) and the possibility of buildup of toxic substances in their main water bodies is a real concern.

Therefore in addition to the use of the coastal water model and estuary model the total loading of toxic substance must be taken into account. There are and will be many sources of toxic wastes which will need to be known and monitored. The total of all these sources should be determined and the mass emission (total loading) used to calculate the allowable loading based on the outflow rates. Except for Lake Superior the inflow concentration of toxic wastes of each lake from the lake upstream must be included in the calculation of total loading.

This whole lake approach may very well prove to be more restrictive on the total allowable toxic loading than the coastal waters or estuarine models especially in the lower lakes. If this proves to be the case, then the permissible loading from each point source will have to be adjusted downward so that the total loading does not exceed

the chronic limits. I am aware that this approach may be utilized under several of the provisions of the Water Pollution Control Act Amendments of 1972 of which the toxic pollutant effluent standards, Section 307(a) is just one.

Section 120.01a Definitions

Many of the affidavits question the definitions of terms and hydrologic restrictions in this section (Petterolf for Michigan, Kinney for Republic Steel Corp., and other industry affidavits). Accordingly the following comments and suggestions are offered to improve this section.

h. "Low flow" - The 7 day, 10 year standard can be viewed as overly restrictive. Variation in stream flow characteristics result in all sorts of special cases. It is better to set some less restrictive low flow definition and require retention of wastes when flow falls below this value. Wastes can then be released when flow rate is high enough to carry wastes without exceeding maximum allowable loading. Specifically a 7 day 1 year low flow is more reasonable, and flow data, if not already available, could be obtained by direct monitoring of flow by the discharger.

The flow of many streams and rivers is presently controlled by dams and reservoirs. Low flow data are often erroneous because the 7-day low flow may have taken from a time prior to the completion of flow control structures.

In other cases policies and practices on flow maintenance have changed. Occasional low flows have also occurred because of plant or mill shutdowns at dam sites. All of these factors can result in unrealistically low flow values.

Recommendation: For streams and rivers with flow control systems, the low flow limit should be taken as that minimum flow which the control organization can guarantee. For streams and rivers without flow control systems, the 7-day, 1 year low flow should be used. If the flow falls below this value, the waste must be retained and released when the flow is sufficiently high to prevent exceeding the desired acute or chronic toxic loading. If, however, the requirements to contain the discharge until satisfactory flow is observed are infeasible, then the use of 7 day once in 10 year low flow should be made a basis for design calculations to provide assurance of compliance and protection of receiving water bodies.

i. "Stream" - reduce retention time to one day or less; otherwise definition is satisfactory.

j. "Lakes and impoundments" - reduce retention time to one day or more.

Justification: Any specific retention time is arbitrary. When flow is interrupted by a barrier and flow gradient decreases to zero, some retention occurs. Retention and interruption of the gravity flow gradient results in mixing and build-up of any pollutant in the body of water to an equilibrium level dependent on the retention time or hydrologic flushing time.(see model) Therefore the lowest practicable retention time is appropriate for the definition.

k. "Coastal waters" - satisfactory

l. "Estuary" - "landward limit of tidal influence" should be replaced by "landward limit where the salinity is 1 part per thousand or less at high tide.

Justification: Tidal influence extends long distances up some rivers and far beyond where the body of water could possibly be considered an estuary, e.g. in the Hudson River the tide extends as far upstream as Troy, New York which is 150 miles from New York Harbor. A generally accepted definition of an estuary is "a semi-enclosed coastal body of water having a free connection with the open sea and within which the sea water is measurably diluted with fresh water deriving from land drainage" (Cameron and Pritchard, 1965).

The one part per thousand salinity limit defines the landward extent of sea water in an estuary in an objective manner and provides a criterion which can be readily determined analytically.

Intermittent and Ephemeral Streams

Kennecott Copper Corp. in the affidavit of Frederick E. Templeton and supported by an attached letter from T. Arnov, U.S.G.S. objects to the application of the proposed toxic effluent standards to the intermittent and ephemeral streams which are common in west and southwest U.S. The U.S.G.S. definitions of these streams are generally accepted. Effluent standards regulations should deal with these types of streams separately. An approach should be utilized which will satisfy the concepts of the acute and chronic toxicological protection parts of the proposed standards employing a case by case determination of the waste streams as evaluated by the Regional Administrator.

Mixing Zones vs. Immediate 1:10 Dilution

The Olin Corporation (affidavit of Louis Rozney), Republic Steel Corp. (affidavit of John E. Kinney), Rohm and Haas Co. (affidavit of L.D. Johnson), State of Michigan (affidavit of C. Fetterolf, Jr.), and others point out that the term "immediate 1:10 dilution" is unrealistic and not attainable. Some of the affidavits also cite the NAS-NAE Report of the Committee on Water Quality and its discussion of mixing zones.

The NAS-NAE discussion and recommendations on mixing zones are general, comprehensive and conservative. The criteria for mixing zones and recommendations require that biological damage be prevented by case definition, by minimizing the area of habitat exposed to mixing processes, and by developing data to show "that the organisms can survive [exposure to the mixing zone] without irreversible damage." These requirements are conservative and difficult to enforce.

Although it would appear that the term "immediate" in the EPA dilution standards is unrealistic, the alternative (NAS-NAE recommendation) is difficult to implement and enforce. Using suitable outfall control devices, a 1:10 diffusion must be attained in the immediate vicinity of the outfall which can be described in terms of effective net discharge diameters or other definable hydraulic terms. This immediate 1:10 diffusion process is more clearly associated with the effluent type standard, while the mixing zone is more appropriately used to implement receiving water quality standards. The latter use requires the necessary determination of an area of the receiving water body that would be knowingly allowed to be in noncompliance with the ambient water quality criteria of the surrounding area.

References

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* I was first the standard deviations in the values ... this was applied to which is derived from the density of water ... of the complete process.
15. Maximum values for constant content of impurities were computed by assuming that all of the measured radium in the sample was contributed by the precipitates.
16. Samples were obtained from the companies: The ... Co., ... Co., ... Co., The Hooker Chemical Co., Simple Co., National Lead Co., and Associated Lead Manufacturers Ltd.; and from I. C. Hubner, G. J. Stekenbroeker, H. Kuhn, W. Bonsted, and A. W. deWaal. We thank these companies and individuals, as well as R. J. Weyl, J. H. Davies, J. G. Fox, I. P. Kaminow, and J. Walker and L. H. Reuter for valuable assistance. The work was carried out in the Nuclear Science and Engineering Corporation, Pittsburgh, in association with the Department of Art Research Projects at Mellon Institute, The National Gallery of Art, Washington, D.C., and ... [illegible] ... further study of the method at Mellon Institute.

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19 October 1966

Natural Displacement of Pollution from the Great Lakes

Abstract. A simplified mathematical model of a lake system indicates that, if the pollution of the Great Lakes were discontinued, the natural flow through the lower Great Lakes would be sufficient to remove about 90 percent of the waste in about 20 years. On the other hand, hundreds of years would be required to displace the pollution from Lake Michigan and Lake Superior.

Approximately 30,000,000 people dump their waste into and drink water from the Great Lakes. During the last few years, the rapidly increasing pollution has become a matter for serious concern. The water in the lower lakes has become offensive, and the cost to the public is tremendous for the increased amount of water-treatment required, the loss of recreational use and the decreased number of fish that are caught (*1*, 2). Multimillion-dollar programs are being proposed to decrease the pollution and to clean up the lake water (2). Many recent publications review the problem of contamination and eutrophication and methods for their control, but none have been found which present quantitative data on the effect of natural displacement by the flow of water through the lakes.

To illustrate the time required for self-purging of the lakes, I have made a mathematical analysis of a simplified model of a lake system. The analysis is predicated upon three assumptions: (i) the precipitation on the lake just equals the evaporation; therefore, the flow rate (R) to and from the lake is the same; (ii) the concentration of pollutants in the streams entering the lake (C_1) is constant; and (iii) the pollutants are added to the lake itself at a constant rate (Q) and are distributed so that their concentration (C_2) is uniform throughout the volume (V) of the lake. A material balance around this lake system gives the relationship of the change in the concentration of pollutants in the lake with time (T):

$$C_2 = C_2^0 \exp(-RT/V) + [C_1 + (Q/R)][1 - \exp(-RT/V)] \quad (1)$$

where C_2^0 is the concentration of pollutants in the lake at the initial time ($T = 0$).

A graph of this function illustrates the rate of buildup of pollutants in an initially uncontaminated lake, $C_2/[C_1 + (Q/R)]$ (curve A, Fig. 1), or the rate of recovery of a contaminated lake when the addition of pollutants is discontinued, C_2/C_2^0 (curve B of Fig. 1). In either case, 90 percent of the final concentration is reached when the volume of water that has flowed through the lake is 2.3 times the volume of water in the lake.

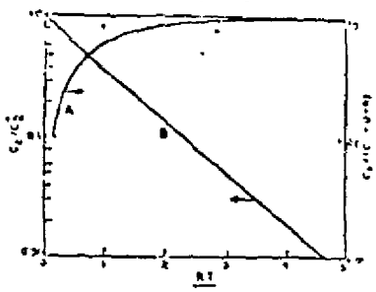

Fig. 1. Effect of water displacement ratio (RT/V) of lake on the concentration of material in the lake. Curve A shows the rate of buildup of pollutants in an initially uncontaminated lake ($C_2^0 = 0$). Curve B shows the rate of recovery of a lake with addition of pollutants is discontinued ($C_1 = 0$ and $Q = 0$).

The lower Great Lakes have a relatively high flow-to-volume ratio. The flow through Lake Erie is 0.38 volume per year (Table 1). The concentration of pollutants would, therefore, reach a nearly steady state in about 6 years if the rate of addition of pollutants to the lake were constant. (The concentration continues to increase largely because of the continuing increase in the rate at which pollutants are added to the lake.) If the addition of pollutants were completely discontinued, 90 percent of the waste would be carried from Erie in about 6 years.

Since the wastes from Lake Erie flow into Lake Ontario, no program for cleaning up Lake Ontario can be effective. Lake Ontario contains about four times as much water as Lake Erie but has only about 20 percent more flow rate. If the addition of pollutants to both lakes were discontinued, approximately 20 years of natural flow would be required to remove 90 percent of the pollution from Ontario.

Because of the low flow rates of the upper lakes, Lake Michigan would require about 100 years to displace contamination by 90 percent to the natural flow; Lake Superior would require more than 500 years.

The mathematical relationship used in these estimates ignores two important factors which influence the contamination of lakes. In actual lake systems the flow of water through the lake is modified by wind and eddy currents, by the bypassing of bays, by chemical uptake, by thermal stratification, and by

Table 1. Data on the Great Lakes system (*1*).

Characteristic	Lake Superior	Lake Michigan	Lake Erie	Lake Ontario
Length (km)	560	490	385	309
Breadth (km)	256	188	91	85
Area (km²)				
Water surface, United States	53,618	58,016	12,898	9,324
Water surface, Canada	28,749		12,768	10,360
Drainage basin land, United States	43,253	117,835	45,620	39,150
Drainage basin land, Canada	81,585		12,224	31,960
Drainage basin land, total	124,838	117,835	58,789	70,488
Drainage basin (land and water), total	207,200	175,860	87,444	90,132
Mean depth (m)	406	281	60	244
Average depth (m)	148	84	17	86
Volume of water (km³)	12,221	1971	458	1616
Average annual precipitation (mm)	736	787	863	863
Mean outflow (liter/sec)	2,067,160	5,012,630	5,550,720	6,626,850
Average retention time of water (yr)	189	30.8	2.6	7.8

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tom topography, and the like. Also, the change in concentration of pollutants is buffered by precipitation and redissolution of materials in bottom sediments, and modified by bacterial action, photosynthesis, and many other factors, in addition to displacement. The assumption of homogeneous dispersal of the pollution in the lake, though physically unrealistic, is a compromise between a fast, purging displacement and a slow diffusion which is the controlling mechanism when there is channeling or stratification. The applicability of the mathematical relationship given above will, therefore, vary with the Lake system. With the lower lakes, the model should apply fairly closely. The upper lakes, however, are deeper, and they are divided by underwater barriers that result in more horizontal and vertical stratification of essentially static water. Also, there is a distribution of intake water around their perimeter rather than from a single upper-lake source. The time for their recovery would likely be even longer than the hundreds of years calculated. Contamination of these upper lakes would, therefore, be a major disaster for which there is no apparent solution.

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References and Notes

1. D. C. Chandler, *Verhandl. Int. Ver. Theor. Angew. Limnol.* 15, 59 (1964); A. M. Beeton, *Limnol. Oceanog.* 10 (2), 240 (1965); C. C. Davis, *Ibid.* 9 (3), 326 (1964).
2. *U.S. News and World Rep.* 59, 58 (12 Dec. 1965).
3. Research supported by the AEC under contract with Union Carbide Corporation.

17 December 1966

Opal Phytoliths in a North Atlantic Dust Fall

Abstract. Minute bodies (less than 50 microns) of microscopic silica, originally precipitated by terrestrial plants, are found together with freshwater diatoms in falls of dust over the ocean. Aeolian transport from Africa can explain the occurrence of similar plant remains in deep-sea sediments of the continental Atlantic as far west as the Mid-Atlantic Ridge.

On 17 January 1965, H.M.S. *Vidal* encountered an intense dust storm northeast of the Cape Verde Islands, approximately 500 km from Africa (Fig. 1). Steaming westward, the ship

Table 1. Frequency distribution (by count) and size ranges of phytoliths, by shape, in *Vidal* dust (see text for explanation of size ranges).

Shape	Incidence (%)	Axes (μ) Long	Axes (μ) Interm.	Figure	Classification (3)
Rod	30	7–60	2–25	2, f and g	*Lithostylidium serra* *L. amphiodon* *L. laeve* *L. curvatus*
Dumbbell (with double swelling, batonnet)	10	10–22	6–18	2, e–	*L. clepsammidium* *L. formica*
Barrel	14	8–14	6–12	2h	*Lithesphaeridium irregulare*
Capstan and hourglass	6	6–18	5–10	2, a and b	*Lithostylidium biconcavum*
Nondescript	40	4–30	3–20		

remained within the storm for more than 30 hours; during daylight the sun was completely obscured. A sample of the dust, recovered from a compressor intake screen (*1*), consisted primarily of clay and silt-sized mineral grains, the two most abundant biogenic components, freshwater diatoms and opal phytoliths (Table 1; *2*), made up about 25 percent of the silt fraction. Freshwater diatoms and phytoliths blown from Africa by the northeast trade winds and recovered at sea were first described in detail by Ehrenberg (*3*). Kolbe (*4*) later reported their occurrence in Atlantic deep-sea sediments.

It has generally been assumed that diatoms occurring in the dust are swept up by the wind from desiccated beds of swamps, lakes, and streams, or from older diatomaceous deposits in arid regions, but that the phytoliths are released from grasses and carried into the atmosphere by prairie fires (*5–7*). However, the silt fraction of some topsoils is more than 50 percent phytoliths (*8*), which may therefore be a component of any wind-blown sediment

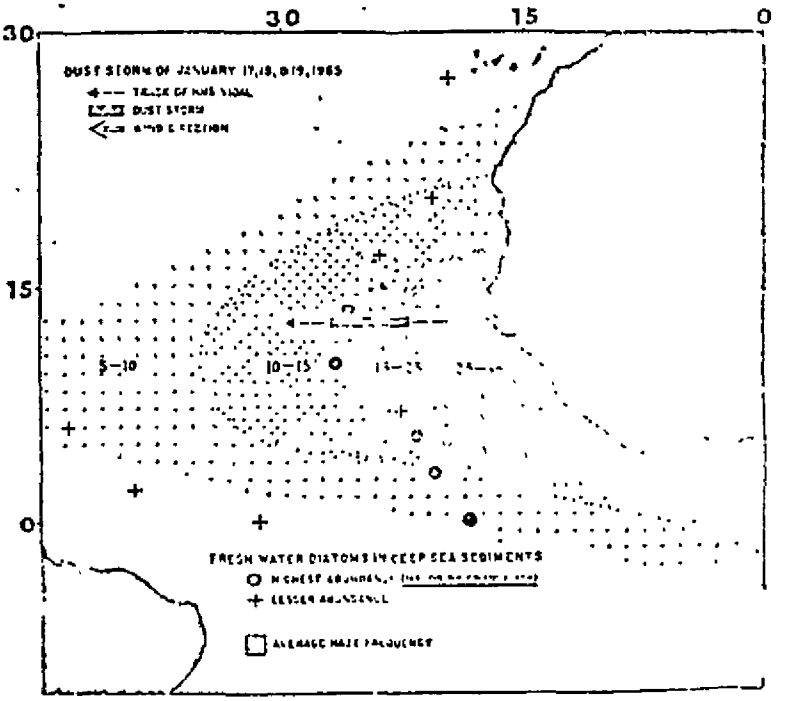

Fig. 1. Phytoliths and freshwater diatoms in a dust storm and in deep-sea sediments (*3*), in relation to the frequency (*1*) and distribution of dust falls (*2*) and to significant transport of dust to the Atlantic by the northeast trade winds (*3*). Phytoliths are associated with freshwater diatoms both in sediment cores and in the air samples recovered during the storm by H.M.S. *Vidal*.

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MIXING PROCESSES IN GREEN BAY

W. F. Ahrnsbrak and R. A. Ragotzkie

Contribution #2 of the Marine Studies Center
The University of Wisconsin–Madison

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880-890. Internat. Assoc. Great Lakes Res.

MIXING PROCESSES IN GREEN BAY[1]

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Abstract. A one-dimensional diffusion model based on the principle of conservation of mass is applied to Green Bay. Observed diffusivities are compared with those predicted on the basis of seiche activity and that mechanism is shown to be primarily responsible for the observed concentration field. Diffusivities in the vicinity of Long Tail Point are on the order of 0.25×10^6 cm^2/sec with an abrupt jump to 1×10^6 cm^2/sec a few km beyond and gradually increasing to 2 to 3×10^6 cm^2/sec in the central part of the Bay. Electrical conductivity and light transmissivity are used to observe the distribution of Fox River water in the Bay. Highest concentration gradients (10%/km) were found in the vicinity of Long Tail Point and along the eastern shore of the southern end of the Bay. No appreciable transverse gradients were found in the mid and northern portion of the Bay. (Key words: Diffusion; pollution; seiche; Green Bay.)

INTRODUCTION

Green Bay (Fig. 1), located near the northwest corner of Lake Michigan, is an elongated bay, approximately 190 km long, with an average width of 22 km. Its principal axis is oriented in a NNE-SSW direction. The Bay, including Big and Little Bay de Noc, has an area of 4212 km^2, a volume of 70 km^3, and a mean depth of 17 m (E. J. Fee, personal communication). No sill separates the Bay from the Lake. Numerous streams and rivers, tributary to Green Bay, are polluted to varying degrees. The largest of these rivers is the Fox, entering the Bay at the city of Green Bay at the south end of the Bay. The Fox River drainage basin has an area of 6520 sq. mi. (16,691 km^2) (Wis. Dept. Nat. Res., 1968a) and the River has a 71 yr. average annual discharge rate of 120 m^3/sec at a point 10 miles upstream from the mouth (U.S. Geological Survey, 1967). E. J. Fee (personal communication) has shown that the Fox River is a major source of domestic and industrial wastes for lower Green Bay.

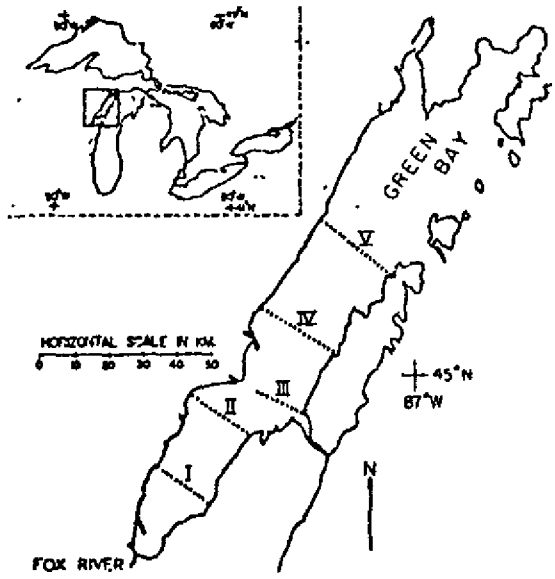

FIG. 1. Location and configuration of Green Bay, Lake Michigan. The dotted lines are locations of cross sections along which measurements for this study were taken.

[1]Contribution No. 2 of the Marine Studies Center, University of Wisconsin - Madison. This research was supported under the University of Wisconsin Sea Grant Program.

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Under the University of Wisconsin Sea Grant Program a broad inter-disciplinary research program is being undertaken on Green Bay. As a part of that program, this paper is addressed to the problem of the rate of dispersal and transport of the waters entering the southern end of Green Bay.

Green Bay can be treated as a one-dimensional system. Pollutants entering the Bay at the south end must either be dispersed longitudinally and ultimately be exported to Lake Michigan, or accumulate—the familiar estuary mixing problem. It has been suggested (Ragotzkie, Ahrnsbrak and Synoviec, 1969) that bays of the Great Lakes can, in some ways, be considered analogous to coastal estuaries of the oceans, and much that is known about estuaries can be applied to bays of the Great Lakes. However, the primary physical mechanisms effecting the dispersal and transport of pollutants may be quite different from those acting in a tidal estuary. Though astronomical tides are lacking, seiches provide an analogous but more complex forcing mechanism for horizontal water movement, and density gradients are entirely due to thermal effects with no salinity contribution.

Csanady (in press) has presented a comprehensive description of the physical factors involved in the dispersal of pollutants in the Great Lakes, and has illustrated quantitative models of small and large scale effluent plumes. Consideration is given, however, to effluent plumes which have free access to an open lake and the geometric constraints imposed in the case of Green Bay are not considered.

Beeton, Smith and Hooper (1967) applied a modified tidal prism model to calculate flushing time for Saginaw Bay, although they did not discuss the physical mechanisms responsible for the observed values.

In addition, some other studies have been done on flushing rates of bays of the Great Lakes. Bryson and Stearns (1959) present an analysis of seiche flushing of South Bay, Manitoulin Island, Lake Huron, in which that mechanism is shown to yield a flushing time of 10 days. Ragotzkie et al. (1969) discuss seiche flushing and flushing induced by wind-induced fluctuations of the thermocline in Chequamegon Bay, Lake Superior. Values given for flushing times are 20 days for the seiche and 10 days for the fluctuating thermocline. In that study, no attempt is made to evaluate any interaction between the two mechanisms, and in both works the weakness of the prism model is present in that complete mixing inside and outside of the bay is assumed.

Calculation of the effective diffusivities on the basis of the principle of conservation of mass has also been used by Stommel (1953), Kent (1960), Snelleck and Pearson (1961), and others. Kent has found values for the apparent eddy diffusivity of from 1.8×10^6 to 4×10^8 cm^2/sec in the Delaware Estuary and Snelleck and Pearson give a mean value of 4×10^5 and a maximum value of 1.3×10^6 cm^2/sec for South San Francisco Bay. In all of these studies, velocity variations caused by tidal action is the forcing function for the diffusion.

METHOD

Based on the principle of conservation of mass, effective longitudinal diffusivities are calculated using a one-dimensional diffusion model for Green Bay.

The following simplifying assumptions are made:

1) The Fox River is the only source of pollutants for Green Bay,
2) The concentration field of Fox River water is a steady state situation (i.e., $\partial c/\partial t = 0$); and,
3) Fox River water is considered to be a conservative variable.

Inherent in the use of the one-dimensional model is the assumption that

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TABLE 1. Average discharge rates of water, suspended solids, and chlorides for 4 rivers entering the southern lobe of Green Bay.

River, Location	Discharge Rate ($m^3 \cdot day^{-1}$)	Suspended Solids: Concentration ($mg \cdot l^{-1}$)	Suspended Solids: Net Transport ($kg \cdot day^{-1}$)	Chlorides: Concentration ($mg \cdot l^{-1}$)	Chlorides: Net Transport ($kg \cdot day^{-1}$)
OCONTO at Oconto	2.35×10^6	9.8	2.3×10^4	6.7	1.57×10^4
MENOMINEE at Marinette	8.96×10^6	5.3	4.75×10^4	1.4	1.25×10^4
PESHTIGO at Peshtigo	2.25×10^6	5.6	1.26×10^4	0.6	1.35×10^4
FOX at Green Bay	11.3×10^6	17.1	19.3×10^4	12.3	13.9×10^4

lateral and vertical mixing take place sufficiently fast that in these dimensions the Bay is essentially "filled," and that further diffusion in these dimensions is constrained by the physical boundaries. This assumption will, in the section on results, be shown to have been valid throughout much of the Bay.

The justification for the assumption that the Fox River is the only source of pollutants entering the Bay can be seen in Table 1. The four significant rivers entering the southern two thirds of the Bay are given with their average discharge rates from United States Geological Survey (1967) records, the average (for the years 1961-1968) concentrations of chlorides and suspended solids (Wisconsin Dept. Natural Resources, 1965, 1968b), and the net transport of those pollutants, calculated as the average concentration times the average discharge rate. Based on these net flows, it can be seen that as a pollutant source, the Fox River is nearly an order of magnitude larger than the sum of the other three rivers.

While the steady state assumption may not be valid for a particular instant, in terms of averages over a two month period the assumption may safely be made. R. F. Modlin (personal communication) has calculated the residence time for Fox River water in lower Green Bay to be approximately 40 days, the same order of magnitude as the period over which data for this study have been collected.

To validate the conservative assumption for Fox River water itself, evaporation and precipitation for the area of the Bay under consideration (that portion south of Section V) was calculated for July, August, and September on the basis of evaporation rates for Lake Mendota by Dutton and Bryson (1962) and for Lakes Michigan and Huron by Folse as discussed by Hutchinson (1957). Average precipitation was calculated from U. S. Weather Bureau records from several stations around the periphery of the Bay. Precipitation minus evaporation (P-E) was then compared with the monthly discharge rates for the total of the four significant rivers indicated in Table 1. Results of the calculations are shown in Table 2. Since the largest value of the ratio of (P-E) to river runoff is about 0.5 percent, changes because of evaporation and precipitation are negligible.

The description of the concentration field was obtained from a number of synoptic surveys in which soundings of temperature, electrical conductivity, and light transmissivity were taken at one meter vertical increments at a number of stations along several cross-sections of the Bay. (Fig. 1). All measurements were taken *in situ*. The temperature and conductivity measurements were made using a Whitney underwater temperature-conductivity meter, and the

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TABLE 2. Evaporation, precipitation and drainage for the southern lobe of Green Bay. Evaporation estimates based on values presented by Dutton and Bryson (1962) are indicated by (D & B). Estimates based on values from Folse (1929) as discussed in Hutchinson (1957) are indicated by (F in H). The area of the Bay under consideration is 2290 km².

	July	August	September	
Evaporation from Lake Mendota (from Dutton & Bryson)	12.7	12.7	11.9	cm.
Evaporation from Lakes Michigan and Huron (from Folse in Hutchinson)	6.7	4.8	3.5	cm.
Total Evaporation for Bay (D & B)	2.79	2.79	2.62	$\times 10^8 m^3$
Total Evaporation for Bay (F in H)	1.47	1.06	.77	$\times 10^8 m^3$
Precipitation (from U.S.W.B.)	8.6	8.3	7.6	cm.
Total Precipitation for Bay	1.89	1.83	1.67	$\times 10^8 m^3$
River Drainage (4 rivers)	2.2	1.6	1.7	$\times 10^8 m^3$
Total Precipitation—Total Evaporation (D & B)	-.9	-1.0	-.9	$\times 10^8 m^3$
Total Precipitation—Total Evaporation (F in H)	.42	.77	.90	$\times 10^8 m^3$
$\frac{P - E}{\text{Drainage}}$ (D & B)	-4.09	-6.25	-5.29	$\times 10^{-3}$
$\frac{P - E}{\text{Drainage}}$ (F in H)	1.91	4.81	5.29	$\times 10^{-3}$

transmissivity measurements were made with a Hydro Products model 410-BR transmissometer using a model 411 10 cm. underwater sensor for all measurements.

Using only one boat, three consecutive days were required to conduct a complete survey. The cross sections in the northern portion of the Bay (IV,V) took two to three hours to complete with the shorter sections in the southern portion of the Bay (I,II) requiring somewhat less time. The surveys in the head of the Bay (Figs. 4,5) took the greater portion of a day to complete.

Electrical conductivity is a function of the amount of material dissolved in the water and transmissivity—concentration relationship is satisfactorily described by an exponential function (Fig. 2).

The basis for calculating the effective diffusivities is the principle of conservation of mass, which can be expressed at any cross section as:

$$F = AK_x \frac{\partial c}{\partial x} + AUC \qquad (1)$$

where

F = flux through the cross section
A = cross section area
K_x = diffusivity
C = concentration of transported substance
U = mean current velocity
X = spatial co-ordinate along the Bay

It was assumed that F is equal to Q, the discharge rate of the Fox River. It was further assumed that other than the lakeward flow of River water, there exists negligibly small mean circulation in the Bay so that this advection is

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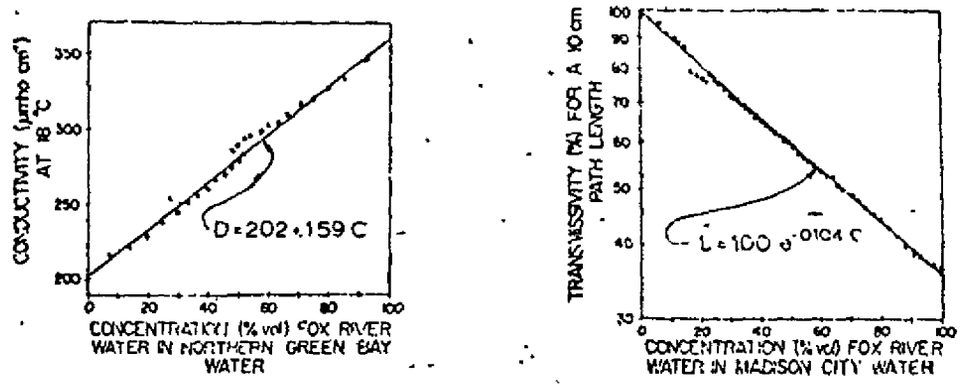

FIG. 2. Empirical relationships between concentration (C) of Fox River water expressed as percent volume, and electrical conductivity (D) and between concentration and light transmissivity (L).

negligible. Since UAC = QC, the ratio of advective effect due to River water transport to total transport is the same as the ratio of River water to Lake water (the units of concentration used in this paper). Therefore, in the regions closest to the mouth of the River, where this effect is the greatest, it is no larger than a factor of two. Lakeward the effect decreases. Effects of this order, on numbers of order 10^4 are considered negligible. Hence the working equation for calculating the diffusivities is

$$Q = AK_x \frac{\partial c}{\partial x} \tag{2}$$

or

$$K_x = \frac{Q}{A \frac{\partial c}{\partial x}} \tag{3}$$

Certainly short-term circulation patterns exist which transport Fox River water lakeward. These are implicitly included in the calculated diffusivity values since measurements of actual gradients were the basis for these calculations, and as noted in the discussion of the concentration field no systematic cross-Bay gradients were observed.

SUSPENDED SOLIDS

Moore and Meyer (1969) have found that a significant portion of the area of Green Bay has become shallower by two to four ft. (0.6 to 1.2 m) in the past 19 yr. Based on that study, the Wisconsin Department of Natural Resources (1965, 1968b) data shown in Table 1, and the description of sediment types existing in Green Bay by A. Hall (personal communication), a correction factor dependent on distance from the mouth of the Fox River was derived. This correction factor was then applied to the concentration of Fox River water indicated by the measured transmissivity values.

It is possible that some re-suspension of sediments sometimes occurs in the very shallow area south of Long Tail Point. However, no diffusivities were calculated for this area. In the central portion of the Bay no marked increases in turbidity were observed following episodes of strong winds and associated

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wave action. Although some sediment movement may occur near the bottom, no further correction for this effect was made. The correction factor used for the effect of settling out of sediments had very little effect on the calculated diffusivity values.

RESULTS

Examples of measured lateral and vertical profiles of the concentrations of Fox River water in two of the cross sections are shown in Fig. 3 and Table 3 gives cross sectional averages of concentration (percent volume) based on transmissivity and conductivity for all cross sections for each survey. In addition to the cross sections, on several occasions an intense survey (approximately 75–100 soundings) was made of the southern 15 km of the Bay. Examples of the results of two of these surveys are shown in Figures 4 and 5.

Using Equation 3, diffusivities were calculated from the one-dimensional model for the concentration fields of Fox River water based on both conductivity and transmissivity. Results for two of the surveys are shown as a function of the distance from the mouth of the Fox River in Fig. 6.

DISCUSSION

Concentration Field

The concentration field of Fox River water in Green Bay is characterized by extremely high concentrations south of Long Tail Point (Fig. 4), with values

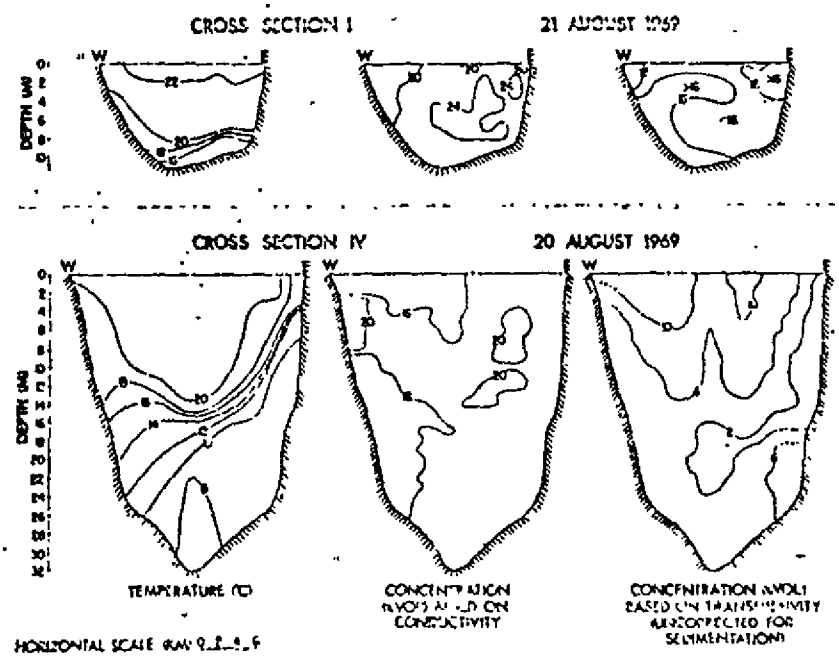

FIG. 3. Temperature (°C) and concentration (percent volume) of Fox River water based on electrical conductivity and light transmissivity for Cross Section I on 21 August and Cross Section IV on 20 August 1969. For locations of cross sections see Fig. 1.

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TABLE 3. Cross sectional average concentrations (percent volume) of Fox River water, based on electrical conductivity and light transmissivity. For cross section locations see Fig. 1.

Date	Section Number	Concentration (% volume) Based on Conductivity	Based on Transmissivity Uncorrected for Sedimentation	Based on Transmissivity Corrected for Sedimentation
15 July	V	9.7	-	-
17 July	IV	14.8	1.2	1.5
29 July	I	28.7	28.4	29.5
29 July	II	18.7	9.5	10.6
18 Aug	III	15.7	4.2	4.9
20 Aug	IV	17.4	4.9	6.3
20 Aug	V	6.9	1.7	2.4
21 Aug	I	22.3	15.5	16.1
21 Aug	II	15.5	7.8	8.8
17 Sept	I	3.3	16.6	17.3
17 Sept	II	6.4	10.1	11.3
18 Sept	IV	3.9	3.7	4.8

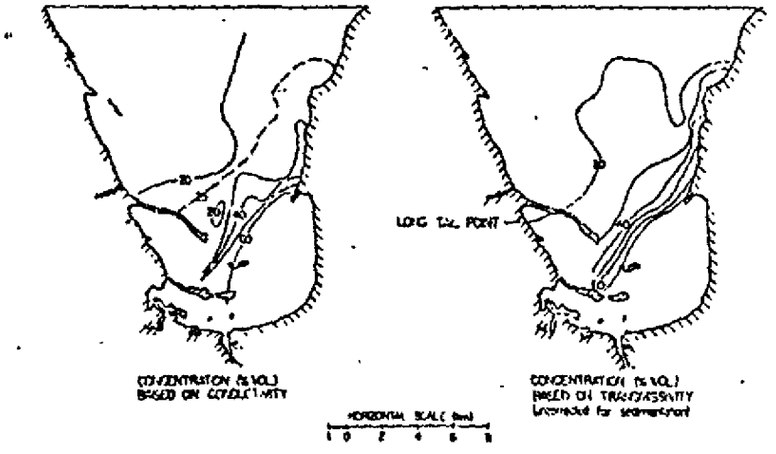

FIG. 4. Surface concentrations (percent volume) of Fox River water in the head of Green Bay on 22 August 1969. Values shown are based on electrical conductivity and light transmissivity.

ranging from 50 to 80% by volume of Fox River water (Figures 4 and 5). Under southerly winds, a tendency for a tongue of relatively high concentrations, 30–40%, of Fox River water can be identified extending northward along the east side of the Bay approximately 15 to 20 km. However, under the influence of northerly winds this tongue was not observed.

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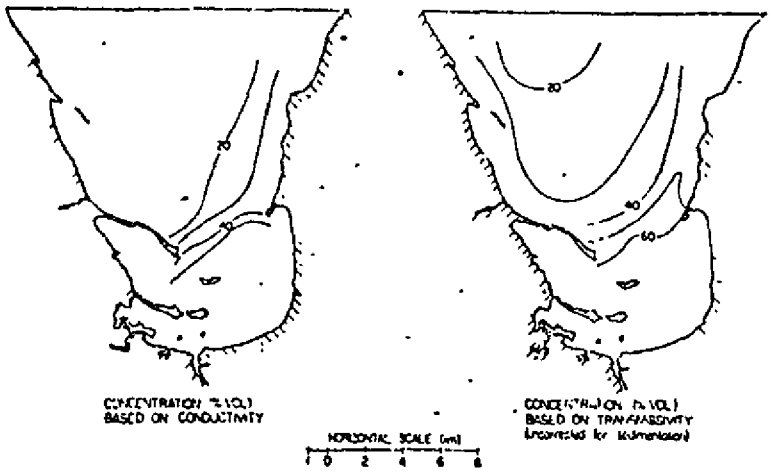

FIG. 5. Surface concentrations (percent volume) of Fox River water in the head of Green Bay on 19 September 1969. Values shown are based on electrical conductivity and light transmissivity.

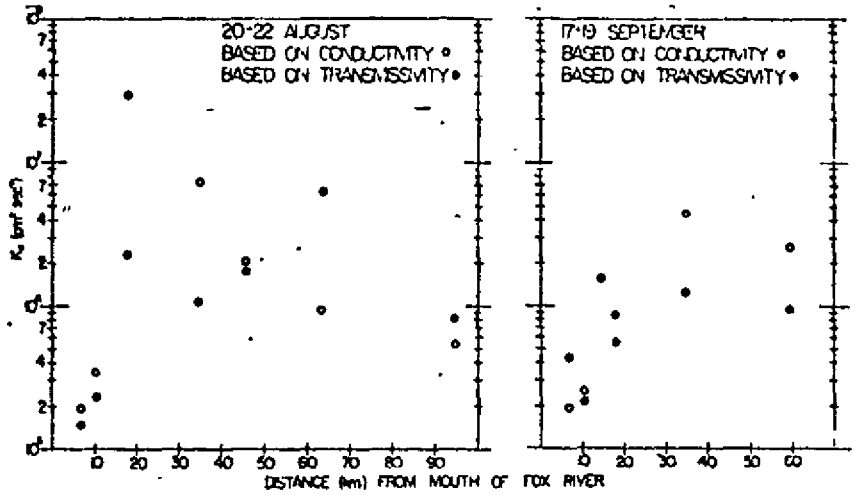

FIG. 8. Observed longitudinal distribution of diffusivities for Green Bay during the periods 20-22 August and 17-19 September 1969. Solid circles represent values from concentration based on transmissivity and open circles represent values from concentration based on conductivity.

In the vicinity of Long Tail Point the concentration decreases very rapidly, a gradient of 30 to 40%/km being observed on several occasions. North of Long Tail Point the concentration decreases much less rapidly to values of approximately 20% observed between 15 and 25 km from the mouth of the River.

North of cross section 1 (approximately 25 km north of the mouth of the River) no systematic cross-Bay gradients were observed (Fig. 3), and longitudinal gradients were very small (less than .5%/km). Concentrations at the northernmost cross section ranged from 0.5 to 10%.

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Diffusivities

The diffusivities in the extreme southern end of the Bay (the southernmost 15 km) were calculated from the one-dimensional model by distorting the effective longitudinal axis of the Bay so that it is perpendicular to the concentration contours. The eight values obtained for the southern end of the Bay, shown in Fig. 6, have a mean value of 0.25 x 10^6 cm^2/sec with a standard deviation of 0.09 x 10^6.

About 20 km north of this, the diffusivity values abruptly increase to approximately 10^6 cm^2/sec or higher (the high value of 39. x 10^6 cm^2/sec in the 20-22 August period seems unreasonable and is probably in error, because of a violation of the steady state assumption). The values then continue to rise, reaching a maximum of 3 to 5 x 10^6 cm^2/sec at about 30 to 40 km. North of this the diffusivity increases slowly, to about 0.7 x 10^6 cm^2/sec at 95 km in the 20-22 August case. This apparent pattern of quite low diffusivity values in the southern end of the Bay, maximum values in the middle region, and a slight decrease near the mouth is present in both the 20-22 August and the 17-19 September case (Fig. 6).

To investigate the mechanisms responsible for the observed diffusivity pattern, the results of a numerical model of Green Bay circulation being developed at the Center for Great Lakes Studies at the University of Wisconsin-Milwaukee (N. S. Heaps, C. F. Mortimer, and E. J. Fee, personal communication) were used. This model takes into account wind stress, free oscillations in the Bay, and a forcing oscillation at the mouth of the Bay due to seiching of Lake Michigan.

Calculation of the predicted diffusivities was based on the assumption that the diffusivity is some function of U', a perturbation velocity, and T, a time scale for the perturbation. From dimensional considerations it can be seen that

$$K \propto U'^2 \cdot T.$$

From inspection of the data the constant of proportionality is seen to be unity in this case.

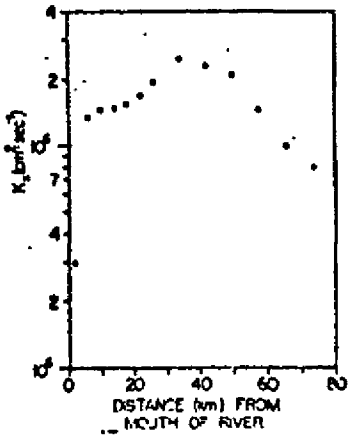

FIG. 7. Predicted longitudinal distribution of diffusivities for Green Bay due to seiche associated water movement, based on numerical model of Green Bay circulation by Heaps et al. (1970).

To calculate the diffusivities, the output from the numerical model of the last 10 hrs of simulated time was used. The longitudinal components at each grid point were averaged over the 10 hour period, approximately the free period of oscillation for the Bay. That average was assumed to be wind generated movement and residuals of the longitudinal components were assumed to be due to seiche activity. The r.m.s. values of the residuals were considered to be the perturbation velocity U' at each point. As a time scale, T, for the seiche perturbations, a value of one half of the free period of the Bay was used. Predicted diffusivities, calculated by this method are shown in Fig. 7.

These results show a value of 0.3 x 10^6 cm^2/sec in the southernmost 3 km of the Bay. Northward the values increase to 1.3 x 10^6 cm^2/sec by a distance of 6 km, further increasing to a maximum of 2.5 x 10^6 at 35 km and then decrease to 0.8 x 10^6 cm^2/sec at a distance of 75 km.

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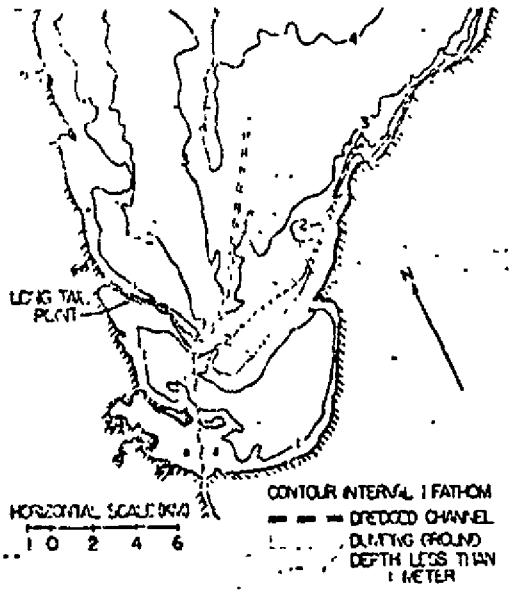

FIG. 8. Bottom topography of head of Green Bay (after U.S. Lake Survey Chart No. 703). Contour interval = 1 fathom.

The agreement between these independently predicted values and the observed results is very good. The most significant discrepancy between the two is the distance from the mouth of the River at which the abrupt increase in diffusivity occurs.

The most probable reason for this discrepancy is that the grid on which the model is based is too coarse to resolve the effect of Long Tail Point and the bar which seems to be its extension (Fig. 8). With the exception of a small area near the dredged shipping channel, depths of the water over this bar are less than one meter. It is postulated that the Point and bar are effectively the end of the Bay for these large scale motions (seiches and larger). Therefore, considering the mouth of the River as the outfall site for the effluent is an error, and the effective discharge of Fox River water into Green Bay takes place in the vicinity of Long Tail Point and the bar which is its extension.

Subtracting 6 km (the distance from the mouth of the River to Long Tail Point) from the distance in Fig. 6, or displacing the origin by that amount brings the two sets of data into good agreement.

CONCLUSIONS

1. The very southern end of Green Bay, south of Long Tail Point, is severely polluted by Fox River water, with a tendency sometimes being seen for a tongue of polluted water extending up the east shore of the Bay for approximately 20 km.

2. North of Long Tail Point the concentration of Fox River water decreases very rapidly, a value greater than 25% seldom being observed north of 25 km north of the mouth of the River. Northward, concentrations are very low and the effect of the Fox River can be described as small.

3. Diffusivities in the southern most 15 km of the Bay are approximately 0.25×10^6 cm^2/sec. At a distance of approximately 15 km an abrupt increase to approximately 1.0×10^6 occurs, followed by a gradual increase to approximately 3×10^6 cm^2/sec at 30 km. Northward the diffusivities decrease to approximately 0.7×10^6 cm^2/sec at 95 km.

4. Diffusivity values of 0.2 to 0.3×10^6 cm^2/sec in the vicinity of Long Tail Point suggest the existence of a barrier to horizontal mixing in this area, while northward, diffusivities an order of magnitude larger imply that that portion of the Bay is well mixed and the transit of Fox River water is much more rapid.

5. Diffusivities calculated on the basis of a mathematical model of seiche-induced and wind-driven circulation patterns in the Bay agree quite

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closely with those calculated from the concentration gradients of Fox River water. This agreement suggests that the pattern of diffusivity values with the highest one occurring in the central portion of the Bay is primarily caused by the seiche activity.

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[illegible]

SUMMER THERMAL STRUCTURE AND CIRCULATION OF CHEQUAMEGON BAY, LAKE SUPERIOR—A FLUCTUATING SYSTEM

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Abstract. An extensive series of [illegible] and [illegible] Chequamegon Bay, [illegible] closed bay [illegible] Analysis of these data shows [illegible] the circulation [illegible] system in which water movement [illegible] [illegible] Superimposed on this fluctuation [illegible] [illegible] measurements also suggest a weak anticlockwise general [illegible] It is [illegible] that the physical behavior of Chequamegon [illegible] may be characteristic of other semi-enclosed bays of the Great Lakes.

INTRODUCTION

Chequamegon Bay is an elongated bay on the south shore of Lake Superior at Ashland, Wisconsin (Fig. 1). Oriented in a NE-SW direction, the bay is 17 km long and averages 8 km across. It has a surface area of 100 km², a mean depth of 6.0 m and a maximum depth of 20 m. The bottom of the Bay slopes downward from the

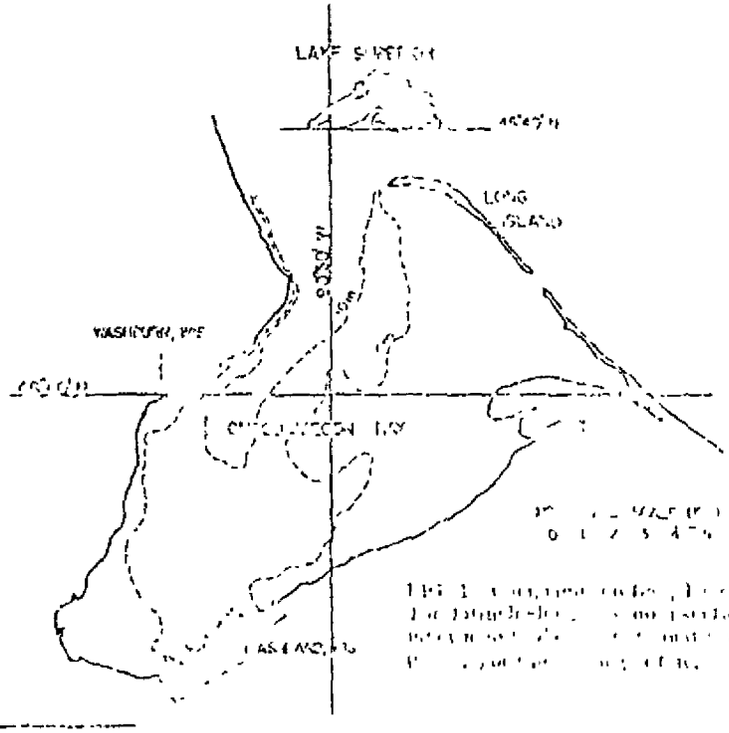

FIG. 1. [illegible] Lake Superior. [illegible]

[1] [illegible]

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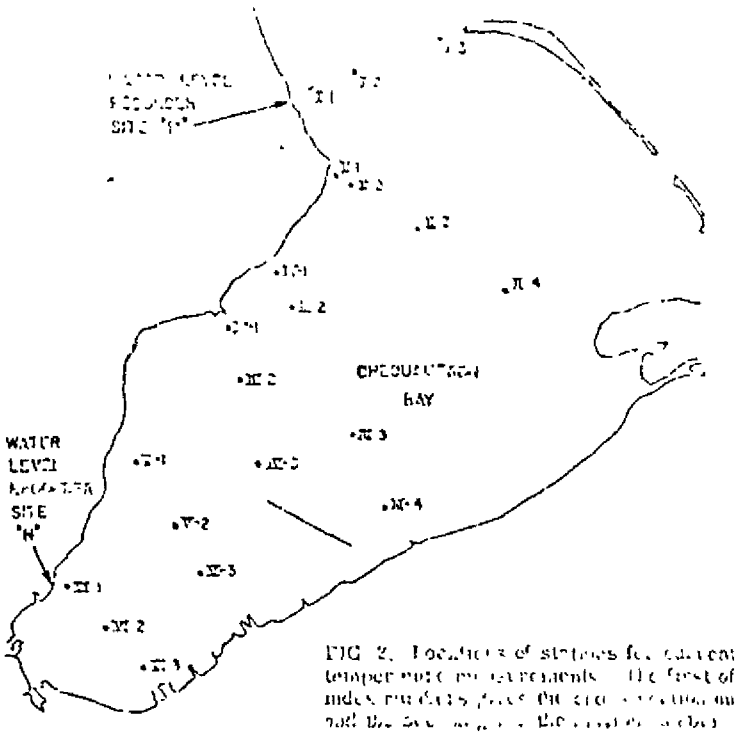

FIG. 2. Locations of stations for current and temperature measurements. The first of the index numbers gives the cross-section number and the second gives the [illegible]

southwest end to its mouth at the northeast end. The south end and the entire east side of the Bay are less than 5 m deep; the deeper water occurs along the west shore. There is no sill separating the Bay from Lake Superior.

The thermal structure, surface oscillations, and circulation of the Bay were studied during the summer of 1968. Special emphasis was placed on the degree and nature of the interaction of the Bay with Lake Superior (Rupotzkie et al, 1969).

PROCEDURES AND RESULTS

Thermal Structure. The three-dimensional thermal structure of the Bay was investigated by repeated temperature measurements along vertical sections oriented across the Bay (Fig. 2). At three stations (I-2, IV-2, and V-2) temperature soundings were taken daily for varying periods of time depending on wind and sea state. Analyses of these data in terms of spatial and temporal variations of the thermal structure are shown in Figs. 2-[illegible].

Prior to the on-site field study one airborne infrared survey was flown over the Bay on 4 June to obtain surface water temperatures. Surface temperatures on this date ranged from 13.7° to 21.3°C indicating that it was already well stratified while Lake Superior does not stratify until early July (Rupotzkie, 1966).

Wind speed (stressing) and direction (cross-section) are recorded every [illegible] at the U.S. Coast Guard station at Bayfield, Wisconsin, located 11 miles west of Washburn (Fig. 1). All winds referred to in this study are taken from these records.

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Surface Oscillations. Two water level recorders (Leupold and Stevens, type F) were installed near the northwest (site "M") and southwest (site "N") ends of Chesapeake Bay (Fig. 2). The instruments were mounted on stilling wells consisting of two 50 gal drums wind bond to one with two 1/4 in holes drilled [illegible] of the column. The instruments functioned almost continuously during the period 6 July-25 August.

A sample of the water level records from the two sites "M" and "N" is shown in Fig. 2. Changes in water level are shown as a function of time.

Preliminary evaluation of all of the seiche records shows that oscillations in the water level were occurring continuously in [illegible] the [illegible] period. A representative sample of the records has been visually analyzed for oscillation periods recorded. Periods shorter than 3 hr which were identified are listed in Table 1.

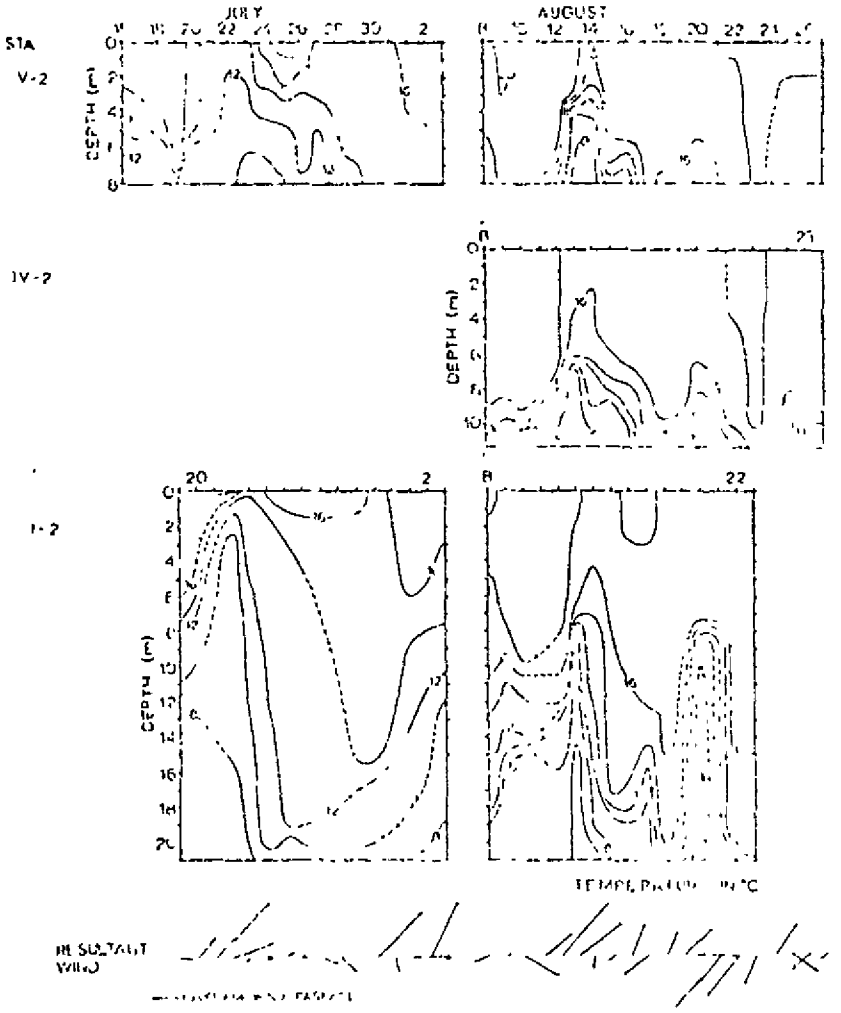

Fig. 2. Results of time series of [illegible] recorded [illegible] at Stations I-2, IV-2, [illegible] during the period 16 July-25 August [illegible] Dashed lines [illegible] interpolation.

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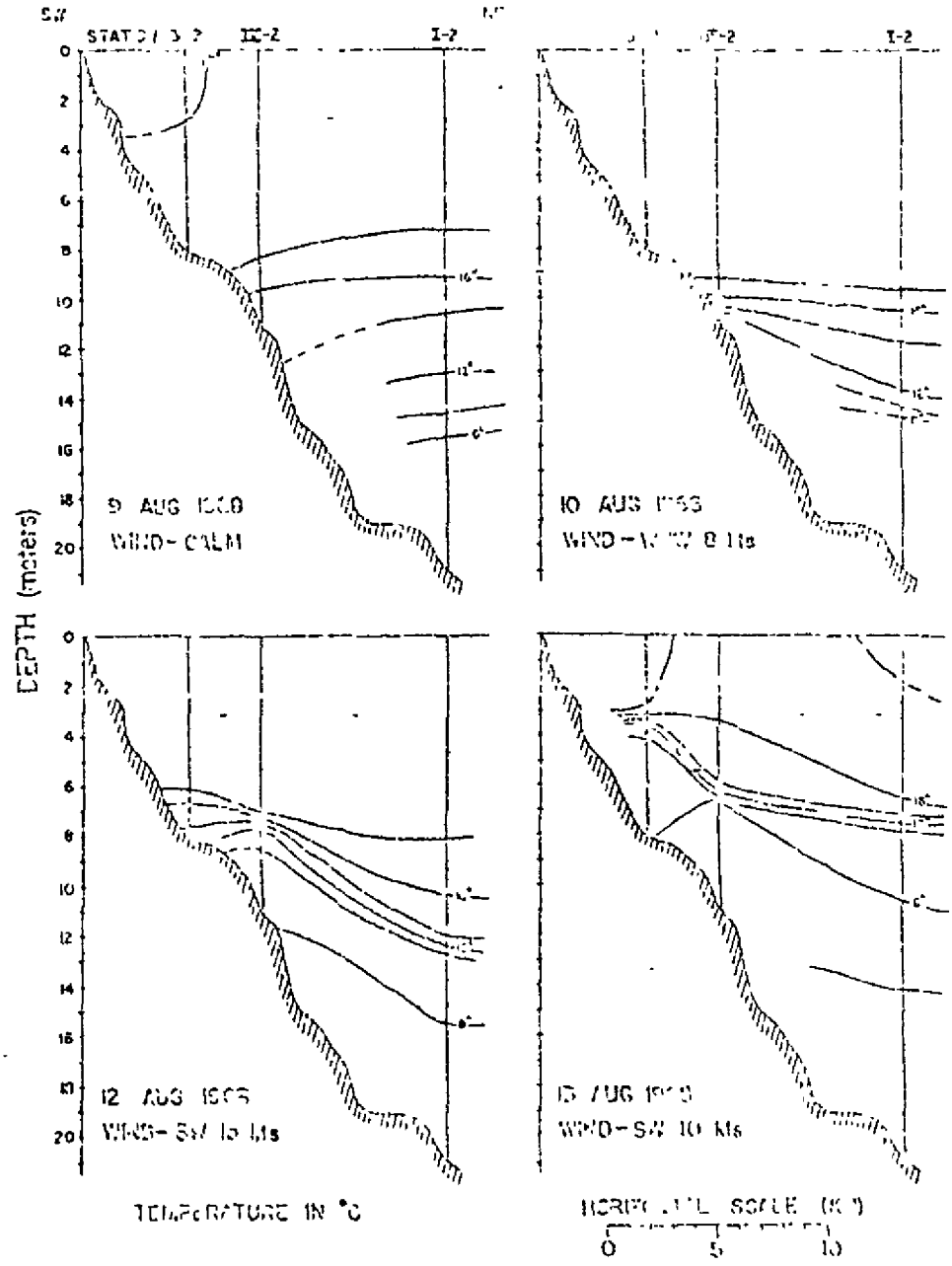

FIG. [illegible] Temperature [illegible] Chocolate [illegible] Station [illegible] Wind indicated [illegible] vector mean wind, obtained from [illegible] Guard Station

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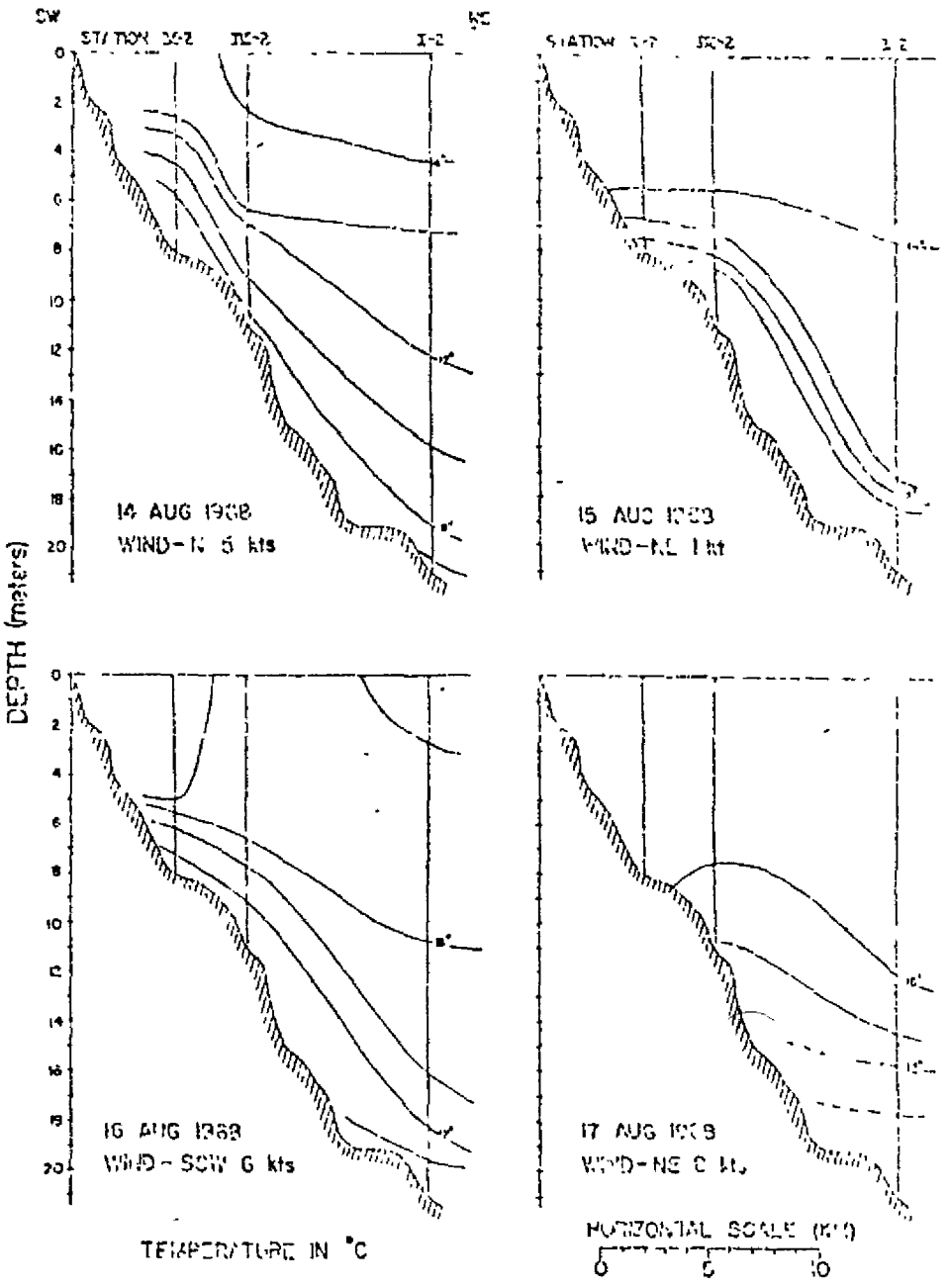

FIG. 5. [illegible] of Cape Kamenny Bay, 13–17 August 1968. Dots indicate [illegible] Isotherms at 1°C intervals. Wind data above each [illegible] vector is based on data obtained from Bayfield Creek Camp [illegible] 2°C. Wind data above each [illegible] vector is based on data obtained from Bayfield Creek Camp [illegible]

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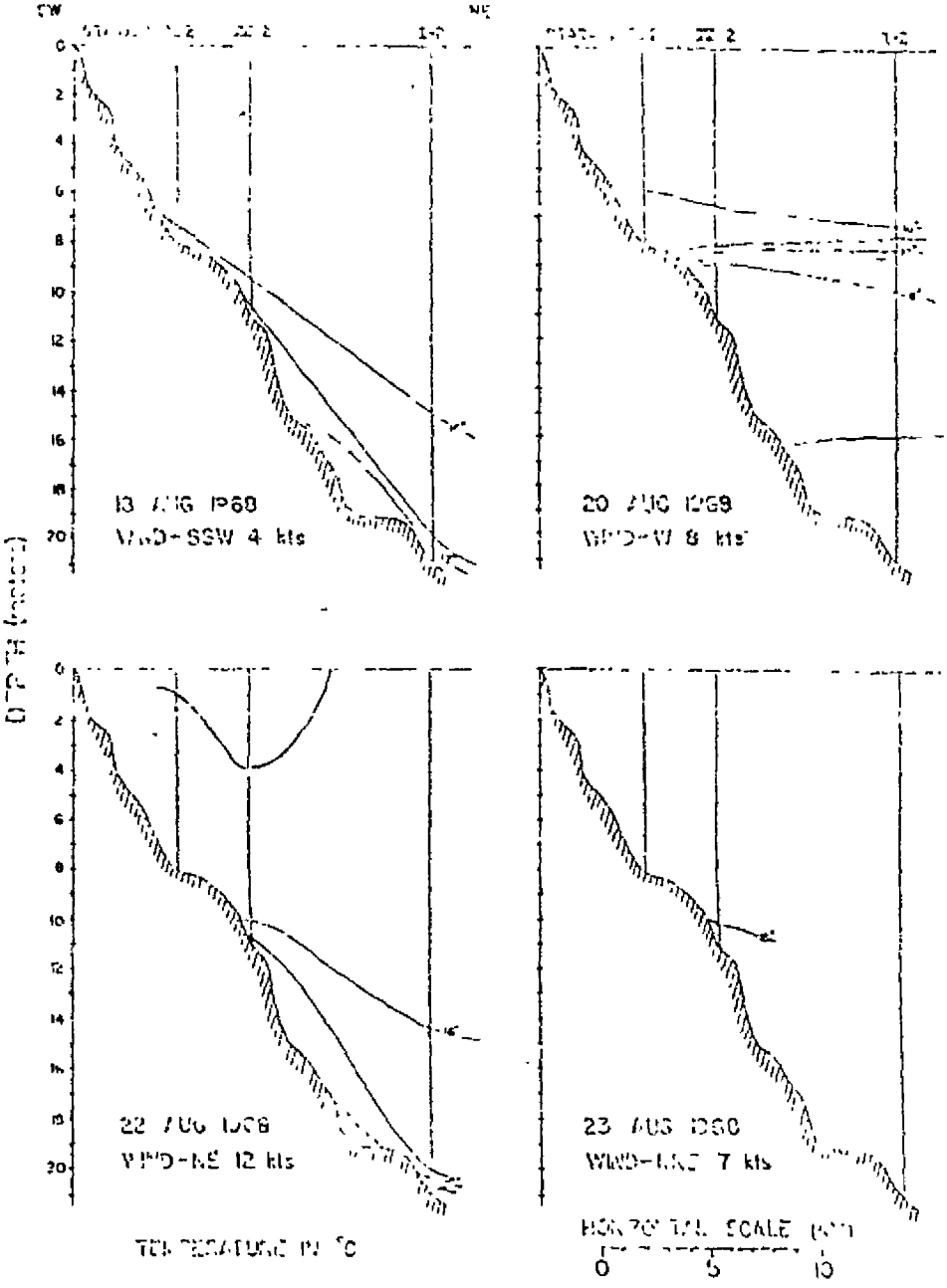
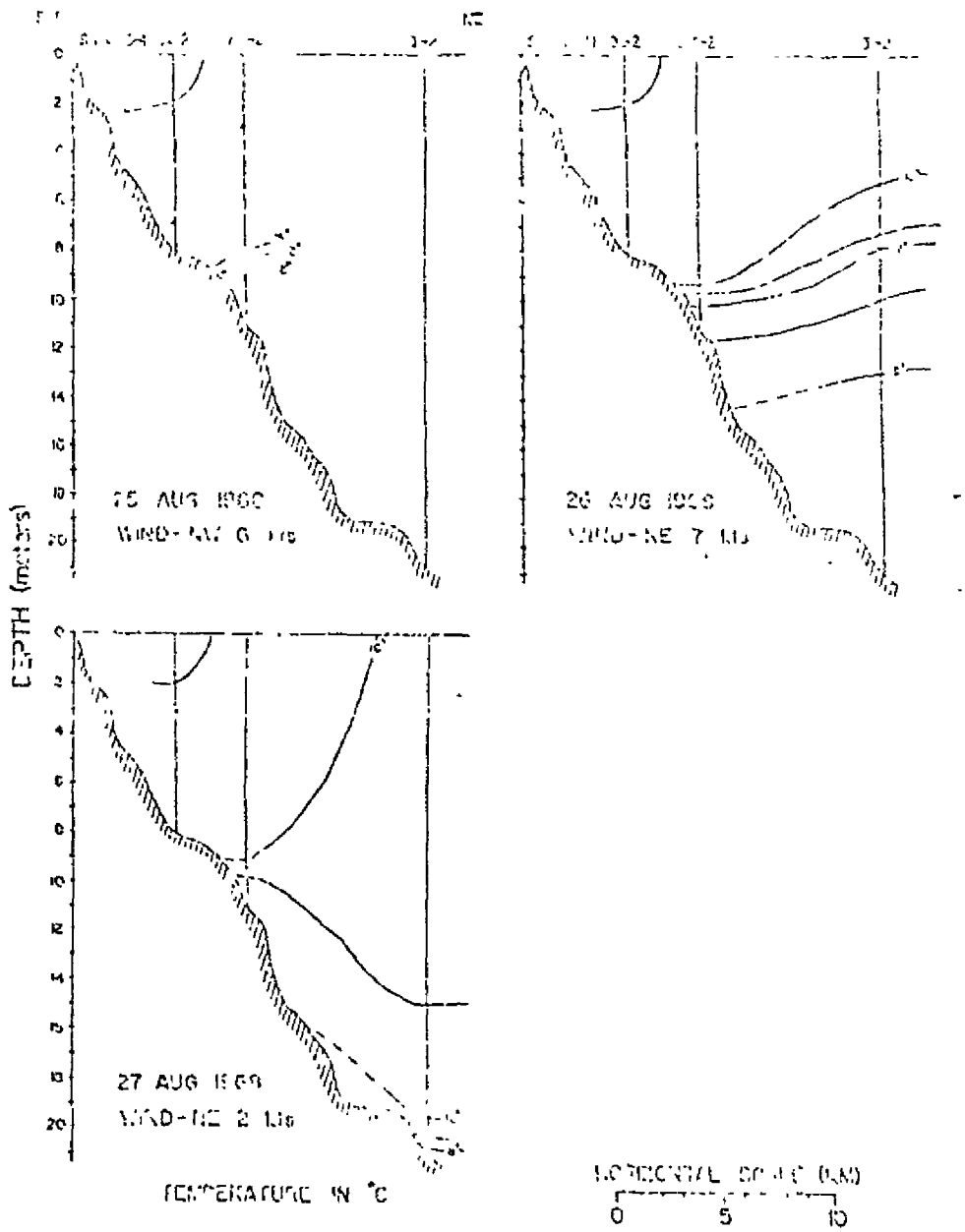

SW
NE
DEPTH (metres)
13 AUG 1968
WIND-SSW 4 kts
20 AUG 1968
WIND-W 8 kts
22 AUG 1968
WIND-NE 12 kts
23 AUG 1968
WIND-ENE 7 kts
TEMPERATURE IN °C
HORIZONTAL SCALE (NM)
0
5
10


DEPTH (meters)
25 AUG 1968
WIND-NW 6 kts
26 AUG 1968
WIND-NE 7 kts
27 AUG 1968
WIND-NE 2 kts
TEMPERATURE IN °C
HORIZONTAL SCALE (KM)
0
5
10

[illegible]

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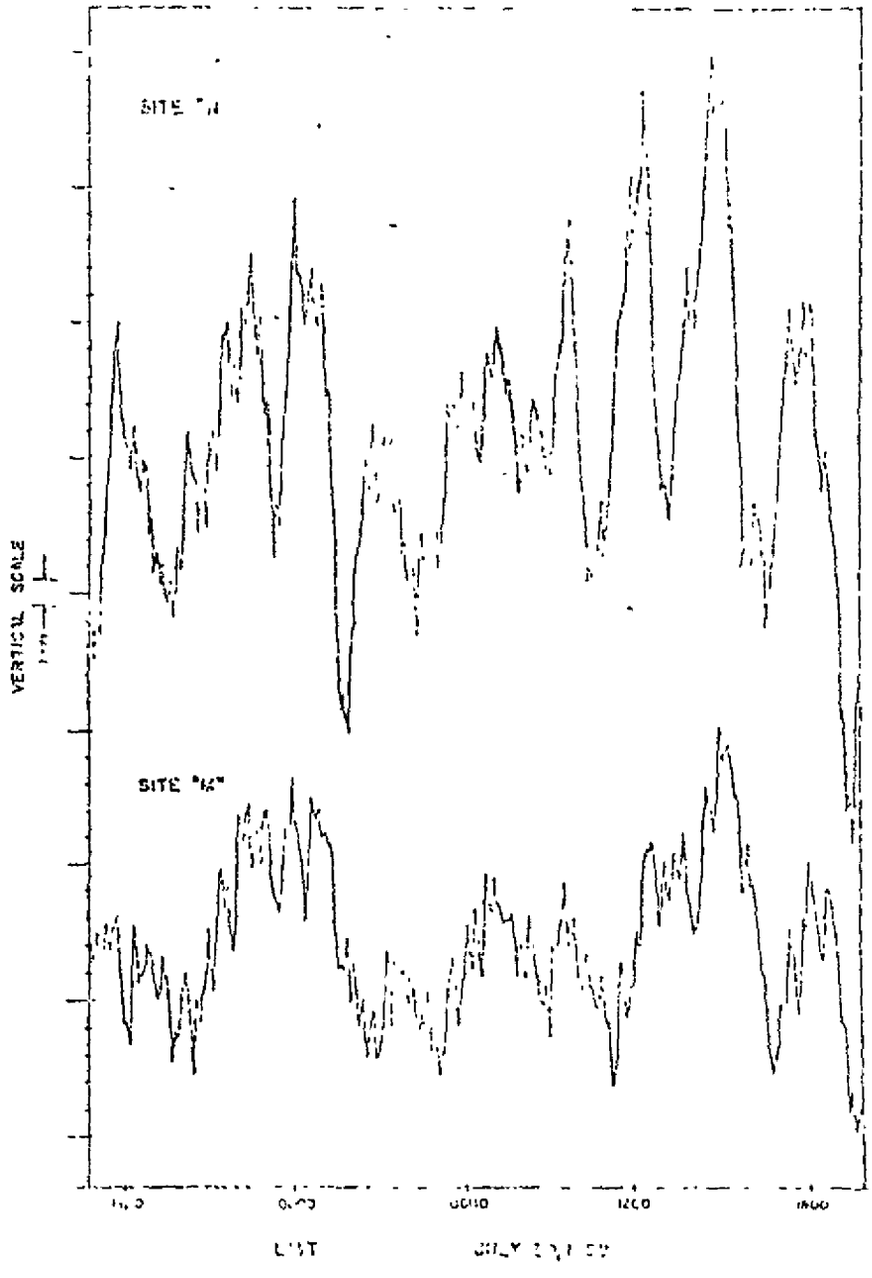

FIG. 8. Sample of the [illegible] for [illegible] on [illegible] [illegible] 25-30 Jul [illegible]

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TABLE 1 Periods of oscillations in the water level of Chequamegon Bay.

n (sec min^{-1})	Theoretical Periods (Merian's formula) (min)	Observed Periods Site "W" (min)	Site "N" (min)
		33	31
3	23	21	21
2	34.5	37	46
1	69	60	62
			96
			130
		161	

Theoretical periods of the various modes of surface oscillation for a basin with the dimensions of Chequamegon Bay were calculated by Merian's formula, $T_n = 2 \cdot \int \cdot (n\sqrt{gh})^{-1}$. These calculated periods are compared with the observed periods in Table 1.

From the values in Table 1 it appears that the uninodal (n=1), binodal (n=2) and trinodal (n=3) seiches all exist in the Bay. Since the wave whose length is equal to the length of Chequamegon Bay has a theoretical period of 69 min, it can be seen that periods of 70 min and lower are due to oscillations which extend around the mouth of Chequamegon Bay.

The frequencies of occurrence of heights of these long waves observed during the entire bi-hour period are given in Table 2. Each wave record was divided into three 2 hr segments and the wave height recorded in each segment was measured. The most frequently occurring wave height at site "W" was 15-20 cm, and at site "N," 10-15 cm.

TABLE 2 Frequencies of occurrence of ranges of vertical oscillation observed in Chequamegon Bay, 6 July - 26 August, 1966.

Range (cm)	Site "W" (% occurrence)	Site "N" (% occurrence)
0 - 5	0	0
5 - 10	1.5	15.2
10 - 15	9.2	34.8
15 - 20	22.9	24.2
20 - 25	20.6	16.7
25 - 30	11.5	5.3
30 - 35	16.0	2.3
35 - 40	1.6	0.8
40 - 45	5.8	0.8
45 - 50	3.6	
50 - 55	2.5	
55 - 60	0.1	

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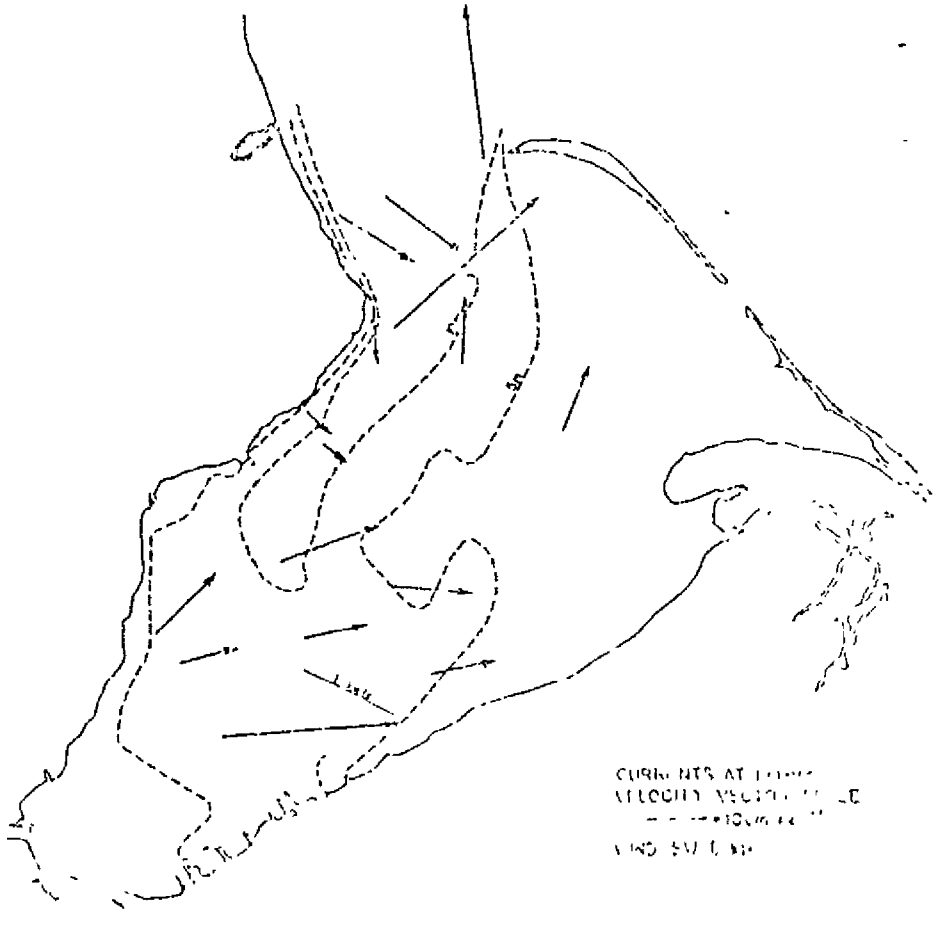
Circulation

1. Currents. All of the current measurements were made using free drogues, a method which has been used in [illegible] (Ellenell, 1967, Carpenter, 1972, Crone [illegible], 1959). [illegible] method [illegible] (19[illegible]). This measurement yields an average [illegible] value of the velocity in the [illegible] coordinate system [illegible], according to [illegible], has an accuracy of 5 to 1[illegible].

Drogues used in this study were [illegible] cross-section of [illegible] aluminum sheet suspended by nylon monofilament attached to plastic bottle floats. Most measurements were [illegible] although [illegible] were used. Current direction was determined by [illegible] magnetic bearings on the float of the drogue [illegible]. Distance traveled by the drogues was measured with a propeller driven [illegible] from the gunwale of the boat, and time elapsed was measured with a stop watch. When currents at more than one depth were measured at a station, several drogues were released in rapid succession, one at each depth, and allowed to drift [illegible]



[illegible]

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ously. The direction, distance and [illegible] of drift [illegible] drogue was determined separately. One boat was used for the [illegible] operation.

[illegible], an [illegible] horizontal [illegible] current is desired; however, the [illegible] required to [illegible] a sufficiently large number of current measurements by [illegible] method precluded reaching this ideal. Compromise was therefore necessary.

Two strategies were employed. [illegible] synoptic surveys were made in which currents were measured at [illegible] depths [illegible] to 16 stations throughout the Bay. These surveys were made on [illegible] July, [illegible] August. The results of the [illegible] surveys are shown in [illegible] in order to [illegible] the effect of the time span required to complete a current [illegible] of the entire Bay, information on the variations of the current with time is necessary. [illegible] of repeated measurements of the current at one or three [illegible] were made on three different days at Station V-2. The results of these [illegible] for 16 July and 2[illegible] August are shown in Fig. 12.

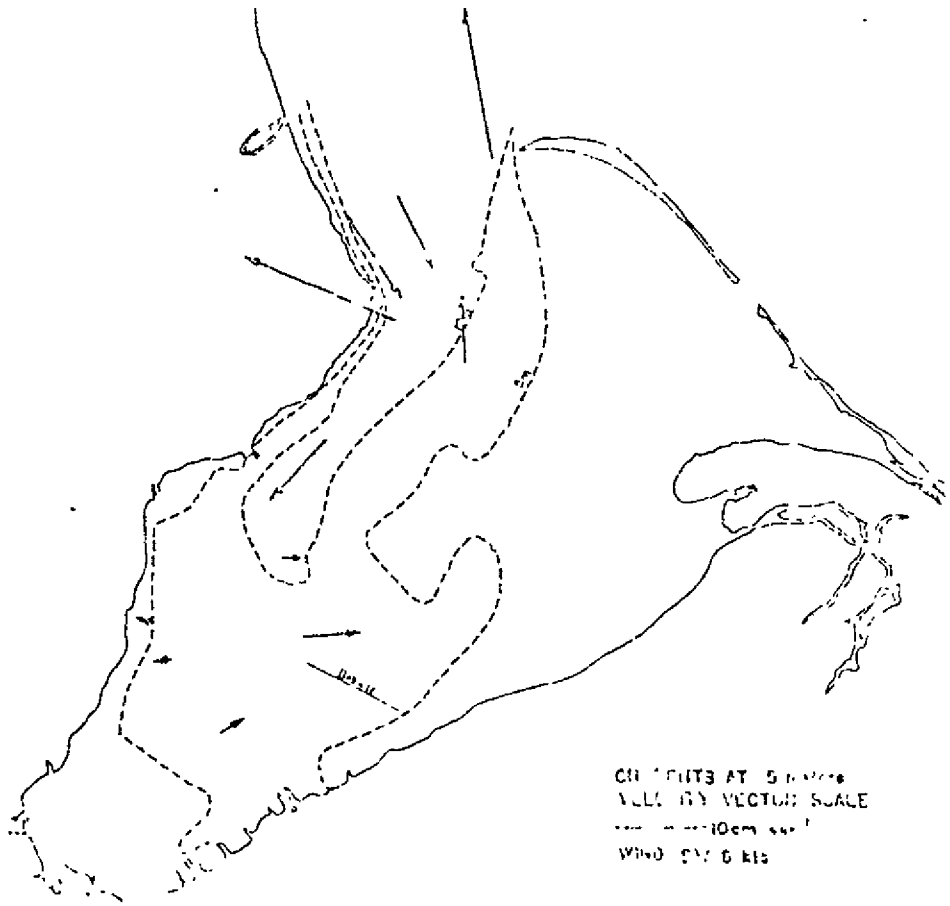

FIG. 10. Currents at [illegible] 1968. [illegible] are [illegible] such that [illegible] at the measurement point [illegible] with length of arrow proportional to current speed. [illegible]

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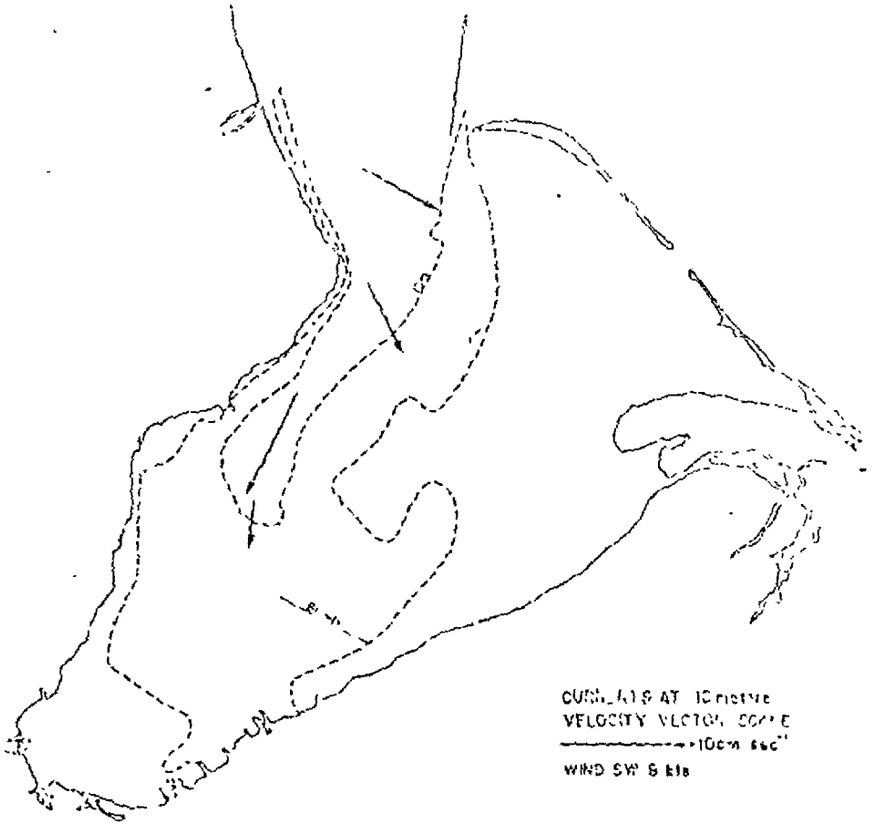

FIG. 10. Currents at 10 m on 1 August 1968. Arrows are positioned such that the base is at the measurement point and are oriented to give the current direction. Length of arrow is proportional to current speed. Vectors have not been rotated.

2. Dye Tracer Experiment. On 10 September 1968 a dye tracer experiment was conducted near Station V-2 using Rhodamine WT, a fluorescent dye with low sorption tendencies. The procedure was as follows: 23 lb of 20% Rhodamine WT in solution was released at depths of 7 m, which is 1 m above the bottom. The movement and dispersion of the resulting dye cloud was observed by pumping the water to the surface and monitoring the fluorescence with a Turner Fluorometer. With this instrument it is possible to measure fluorescence concentrations over a range of five orders of magnitude, and to a precision of 0.1 ppb.

On [illegible] of the experiment the vessel set out at the mouth of the York River and proceeded up the bay at 10 to 12 knots to the mouth of the Rappahannock River and then [illegible] CBI Buoy 1 [illegible] Vessel and dye were then positioned at the release point and the dye was released. Water temperatures ranged from 6°C near the surface to the bottom of 9°C. At 1300 the surface layer was [illegible] vertical temperature gradient slightly to 8°C from top to bottom. The [illegible] and Potomac [illegible] [illegible] a cream colored [illegible] [illegible] [illegible]

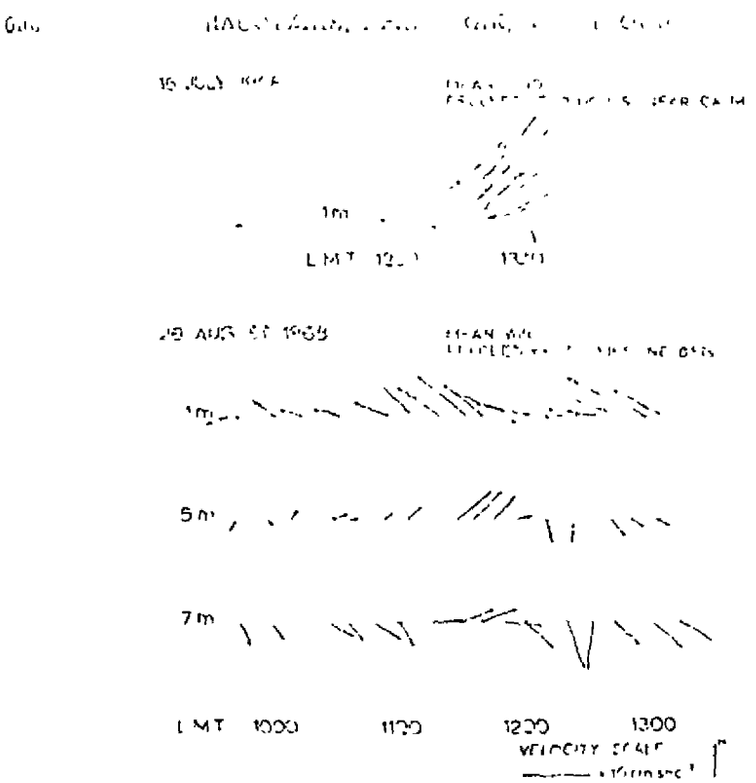

FIG. [illegible] Time series of current measurements made at Station V-2 on 17 July and 26 August [illegible]. Arrows are positioned with the base of the arrow at the time at which the measurement was made. Orientation of the arrow indicates current direction and length of the arrow is proportional to current speed. Wind is heated as from the records of the Bayfield Coast Guard Station.

Two attempts were made to carry out the experiment. The first dye release was made at 1000h. This experiment failed because the dye cloud was lost in the first few minutes. Shortly after this first release, a very small amount of dye appeared at the surface. This was most likely due to the method of release, namely, by pumping the dye solution through a U-tube at the end of a rubber hose, which effectively jetted the dye upward with sufficient force so that a small amount reached the surface. The surface cloud remained small however, and it was apparent that most of the dye stayed well below the surface and out of sight.

The second dye release was made at 1525 also at 7 m below the surface and 1 m above the bottom. The release tube was aimed horizontally and the dye introduced over a period of about 10 minutes order to enlarge the cloud. Approximately [illegible] following this second release [illegible] dye [illegible] close enough to the surface to be observed visually. The cloud was located and successfully followed by fluorometer [illegible] and 190 [illegible].

Current measurements [illegible] were made at the release point [illegible] at the termination point at the end [illegible].

The results of the tracer experiment and [illegible] currents are shown in Fig. [illegible] [illegible] the apparent [illegible] of the dye cloud [illegible] scale. The time lines

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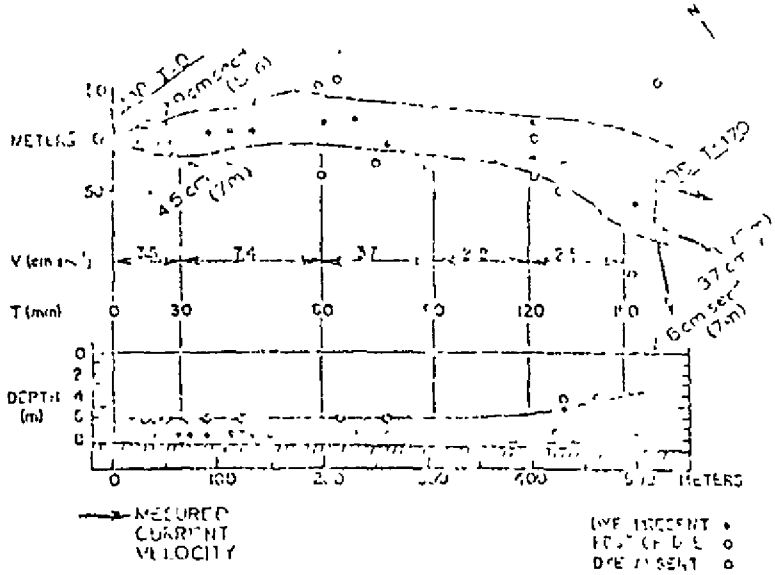

FIG. 13. Plan view and vertical section of trajectory of dye cloud released at Station V-2 on 10 September 1968. Arrows with oblique levels represent velocities measured with [illegible]. The [illegible] based on the position of the leading edge of the cloud. Time shown is time elapsed [illegible] after dye release.

indicate the estimated position of the leading edge of the cloud at 30 min intervals. For each time interval conservative estimates of the velocity of the leading edge of the cloud are given in cm/sec. The agreement between the estimated velocity of the dye and the current velocities measured by free drogues is excellent (Fig. 13). Note that the dye, located between 6 and 7 m depth has an initial velocity of 3.6 cm/sec while the drogue measurements gave velocities of 2.5 cm/sec at 5 m and 4.5 at 7 m or an average of 3.7. Similarly the dye at the end of the experiment then located between 5 and 7 m was estimated to [illegible] at 2.5 cm/sec and the drogue measurements gave velocities of 3.7 and 4.6 at 5 and 7 m respectively.

During the experiment the dye cloud increased in width gradually from 25 m at t + 30 min to 90 at t + 150 min. The dye moved approximately southward which corresponded very well with the drogue measurements at the depth where the dye was located (Fig. 13). The mixing of the dye was limited almost [illegible] to the horizontal plane despite the weak thermal stratification prevailing during the experiment. No dye was detected above the 7 m release level [illegible] 2 hr after the release. In the last 45 min of the experiment the dye was detected at 5 m at two locations, but even then most of the cloud was apparently still at 7 m. Sampling below the 7.5 m level was not possible because of the risk of pumping mud through the [illegible].

[illegible] the difficulty [illegible] the three dimensional [illegible] pumping [illegible] data on the [illegible] of the cloud [illegible] concentration. Therefore, no reliable estimates of dye [illegible] could be [illegible] 2 hr after the release, the [illegible] dye concentration [illegible] orders of [illegible] concentration of the released solution.

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Discussion

[illegible] three [illegible] [illegible] [illegible]

was done by direct... the Bay ... 10 transect... sections ... cent... the ... of ... the ... Bay ... Principal ... of the Bay ... Fraser ... period ... 20-1... ... tidal ... ... inflow of ... ... equal ... to 5... ... ...

Speed (cm/sec)	1		5		7		10		Total		Cumulative %
	no.	%	no.	%	no.	%	no.	%	no.	%	
0 - 2.9	9	9.3	25	38.5	2	7.4	0	0	36	14.3	100.0
3 - 5.9	19	21.1	21	32.3	16	59.3	6	50.1	73	29.1	85.7
6 - 8.9	50	22.3	12	17.8	3	11.8	1	50.6	91	24.3	56.6
9 - 11.9	19	13.1	5	7.2	1	3.7	2	15.1	27	10.7	32.3
12 - 14.9	21	14.5	2	2.9	0	0.0	0	0.0	23	9.2	21.6
15 - 17.9	10	7.0	1	1.4	1	3.7	1	7.7	13	5.2	12.4
18 - 20.9	6	4.2	1	1.1	0	0.0	0	0.0	7	2.8	7.2
21 - 23.9	5	3.5	1	1.1	0	0.0	0	0.0	6	2.4	4.4
24 - 26.9	2	1.1	0	0.0	0	0.0	0	0.0	2	0.8	2.0
27 - 29.9	0	0.0	0	0.0	2	7.4	0	0.0	2	0.8	1.2
≥ 30.0	0	0.0	1	1.1	0	0.0	0	0.0	1	0.4	0.4

These seiche currents superimposed on the wind-driven general circulation of the Bay result in an increased variability in the observed currents. While the interaction of the two kinds of motion makes interpretation of the observed currents more difficult, conclusions drawn from the current surveys are not necessarily invalidated by the interference of seiche induced currents.

The 60-min seiche is accompanied by a vertical displacement of the water surface which is easily detected by the pressure gauges. This is verified by the two time series of current measurements shown in Fig. 12. In the 16 July series a 60-min periodicity of current velocity is readily identified while on 26 August the current fluctuation appears to be dominated by the 140-min oscillation.

The 140-min seiche is a special case. A first order calculation of the wavelength of this seiche indicates that the nodal line lies somewhere near the mouth of Chequamegon Bay. An oscillating horizontal motion induced by the seiche across the nodal line will result in transport of water in and out of the Bay itself. As water moves out of the Bay it can be expected to be entrained in steady, lake current's flowing past the Bay, usually eastward along the south shore of Lake Superior (Saylor and Sherlock, 1968; Saylor, 1970). Then when the return flow occurs across the nodal line, new water will flow into the Bay. In this way a regular flushing action occurs with a period of about 140-min. A first order approximation of the exchange rate was calculated assuming that the surface of the Bay remains plane. The results (Table 4) indicate that a 10 cm vertical oscillation

TABLE 4. Flushing rates due to seiche with nodal line across mouth of Chequamegon Bay

Height (cm) of vertical displacement at Saxon Harbor	Flushing Rate	
	Percent of total volume per cycle (2.6 hrs)	Percent of total volume per day
5	0.25	2.3
10	0.5	5.1
20	1.0	10.3

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[illegible] day. Doubling the [illegible] exchange rate. [illegible] mechanism was [illegible] and [illegible] in [illegible] and Collins (1970).

CONCLUSIONS

The circulation and thermal structure of Chequamegon Bay are governed by (1) the interaction between the Bay and Lake Superior with the continuous intrusion of the Lake Superior water, and (2) the wind. The wind, through [illegible] thermal stratification, develops [illegible] and [illegible] pushes a large proportion of the warm epilimnion accumulated in the Bay eastward to the Lake. This process responds to variable wind intervals, reaching maximum development in the order of one week or less. At [illegible] the activity of the Bay [illegible] probably results in a flushing rate approximately 10% of the [illegible] volume per day. In this way Lake Superior receives a steady supply of warm water which is entrained in the general west to east [illegible] current along the south shore of the Lake (Saylor, 1970).

It is suggested that this pattern is typical of other semi-enclosed bays of the Great Lakes. A similar scheme has been described for Green Bay of Lake Michigan by Noble and Scarpa (1969). Green Bay of Lake Michigan is also known to warm and stratify much earlier than Lake Michigan because the [illegible] through the openings at the north end of the bay are known to be small and variable.

Flushing rates will vary according to the size of the bay, its width opening to the lake, and the presence or absence of a well-developed [illegible] current. In the Bay, it seems likely that flushing will occur due to [illegible] forced oscillations of the surface and the thermocline of the bay.

Bays of the Great Lakes can be considered as somewhat [illegible] estuaries at the oceans. The wind-induced oscillations of the surface and the thermocline of Great Lakes bays [illegible] are [illegible] like the tidally-driven oscillations of an estuary. Thermal [illegible] by surface heating in the Bays produce density gradients during the summer [illegible] to those found in estuaries due to salinity gradients. More specifically the Bay system behaves most like the "moderately stratified estuary" described by Cameron and Pritchard in vol. 2 of *The Sea* (1963). This type of estuary is characterized by the persistence of a vertical gradient (of salinity in the estuary) [illegible] caused primarily by "turbulence resulting from tidal motion". In the Bay case the vertical gradient is thermal and mixing is caused primarily by wind-induced turbulence. In both cases a horizontal gradient persists with more dense (saltier or colder) water occurring seaward.

Although this analogy is not a rigorous one in terms of [illegible] the similarities in behavior between the two [illegible] is known about estuaries could be applied, at least in principle, to bays of the Great Lakes. Both coastal and [illegible] characteristics [illegible] specifically for estuaries. The [illegible] and the essentially [illegible] the incoming water from river to lake via a bay is a specific problem where the estuary analogy could be useful.

CONCLUSIONS

1. Chequamegon Bay is [illegible] and circulation [illegible] Lake Superior [illegible] Bay is [illegible] Lake Superior water.

2. The Bay is [illegible] during the summer season, [illegible] cool deep Lake Superior water.

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2. Exchange [illegible] and [illegible] of the [illegible] of the [illegible] of water surface water [illegible] from [illegible] from [illegible] face [illegible] ing. [illegible] frequency of cyclonic storms.

4. Seiche [illegible] with periods of 12, 2[illegible] 62, and 140 min. Horizontal [illegible] 140-[illegible] current across [illegible]

5. [illegible] and [illegible] and [illegible] first, by [illegible] of the Bay with the Lake is not unusual).

6. The circulation and fluctuations [illegible] probably typical of most semi-enclosed bays of the Great Lakes.

7. On the basis of their physical characteristics [illegible] to coastal estuaries of the oceans.

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AFFIDAVIT OF HUGO B. FISCHER

Introduction

At the request of the Environmental Protection Agency I have reviewed the proposed rules for toxic pollutant effluent standards published in the Federal Register, Vol. 38, No. 247, Thursday, December 27, 1973, pages 35388 through 35395, and the basis for these proposed rules as set forth in Agency "White Papers" supplied to me, and the affidavits of a number of objectors to the proposed rules. The scope of my review is limited to the hydrodynamic basis for the proposed rules, a field in which I am qualified to testify by virtue of education and professional experience as follows: I have received the degrees of B.S., M.S., and PhD. in Civil Engineering, from the California Institute of Technology. I am presently an Associate Professor of Civil Engineering at the University of California, Berkeley; since 1964 I have conducted research continuously on the subject of hydrodynamic mixing processes in rivers, reservoirs, estuaries, and coastal waters, and I am the author or joint author of 38 published works in these fields (see attached list), including a review paper in the Annual Review of Fluid Mechanics; and I am the author of a 250 page unpublished manuscript entitled "Hydrologic Mixing Processes" which is used as the text in a graduate course at the University of California and which covers a broad range of subjects relevant to the hydrodynamic basis for the proposed rules.

I do not represent myself as knowledgeable in the fields of toxicology or physical and biological reactions in water, and I shall confirm my testimony on the Hydrodynamics of Mixing processes.

The proposed rules are based on protecting against the effects of chronic toxicity by means of limiting concentration in the receiving water flow. The assumption is made that within a time span short enough to be less than that necessary for the appearance of chronic effects complete mixing will take place between the effluent stream and the receiving water stream. Hence the standards are based on requiring that the concentration in the combined stream, after complete mixing, be less than that thought to produce effects of chronic toxicity. Whether this is a proper basis for regulation or not is beyond my field of special knowledge; therefore my testimony is based on acceptance of this principle, and is limited to suggesting some changes in the way the receiving flow is computed in estuaries and coastal waters and providing some information on mixing time in streams. I will also offer testimony on the subject of initial dilution for protection against effects of acute toxicity. These areas seem to be those which objectors have questioned in their statements and those which I am best qualified to address.

In his statement, Mr. Mancini (on behalf of American Cyanid), points out that for estuaries which are located near the ocean or gulf, larger mass emission rates than allowed in the proposed rules would provide comparable protection to the environment. This appears to be correct.

Quantification of the Receiving Flow in Estuaries

In an estuary, the quantity of flow available for dilution of the waste stream contains two components, the flow of the tributaries, as defined in section 129.01c(c)(3) of the proposed rules, and an effective flow coming from and returning to the ocean due to the tide. Actually the oceanic flow is progressively diluted as one moves upstream from the mouth of the estuary, but for purposes of analysis one can assume, as sketched in figure 1, a flow of pure ocean origin water coming from the ocean to the point where the effluent is discharged, mixing with the effluent and tributary discharges, and returning to the ocean. When the streams are fully mixed the resulting concentration is given by:

$$M = (Q + Q_e + RP/T)\, c \qquad (1)$$

in which M = discharge rate of material (mass per unit time)
Q = tributary discharge
Q_e = waste stream discharge
R = tidal exchange ratio
P = tidal prism upstream from the waste stream discharge point
T = duration of tidal cycle
c = concentration of the material discharged at rate M

In this equation R is the tidal exchange ratio, defined as the ratio of new ocean water to total tidal flow moving past the discharge point during a flood tide, and RP/T is the effective flow of new ocean water available for dilution.

The tidal exchange ratio may be measured empirically as follows. Let

V_f = total water volume entering the estuary on the flood tide -

V_{fe} = that part of V_f which flowed out of the estuary on the previous ebb

V_o = volume of new ocean water entering the estuary during the flood tide

V_e = volume of water leaving the estuary on the ebb tide

V_Q = volume of fresh river water plus waste stream water entering the estuary during the tidal cycle

s_f = average salinity of water entering the estuary on the flood tide

s_e = average salinity of water leaving the estuary on the ebb tide

s_o = salinity of the ocean water

If the total salt and water contents of the estuary are to remain constant we have

$$s_f V_f = s_e V_e \qquad (2)$$

and

$$V_f + V_Q = V_e \qquad (3)$$

also

$$s_f V_f = s_e V_{fe} + s_o V_o \qquad (4)$$

and

$$V_f = V_{fe} + V_o \qquad (5)$$

The tidal exchange ratio can be defined as

$$R = V_o / V_f \qquad (6)$$

Combining the above equations it is easy to show that

$$R = \frac{S_f - S_e}{S_o - S_e} \qquad (7)$$

and with further manipulation,

$$R = \frac{S_e}{S_o - S_e} \quad \frac{V_Q}{V_f} \qquad (8)$$

By definition $V_Q = (Q + Q_e)T$ and $V_f = P$, so we have

$$R = \frac{S_e}{S_o - S_e} \quad \frac{(Q + Q_e)T}{P} \qquad (9)$$

Substituting this result into equation 1 gives

$$M = [(Q_e + Q) \frac{S_o}{S_o - S_e}]C \qquad (10)$$

The same result may be obtained by an alternate derivation. The salinity at any point in an estuary is a measure of the dilution of the freshwater inflow. Once a waste stream has become well mixed with the freshwater discharge, through cross-sectional mixing, it will have the same concentration as if it had been discharged in the freshwater section. Hence the effective dilution flow of ocean water is the same for the waste stream as for the freshwater, and can be computed from observations of salinity. If Q_o is the discharge of ocean water available for dilution, conservation of mass gives

$$Q_o S_o = (Q + Q_e + Q_o) S \qquad (11)$$

where S is the salinity at a cross section averaged over the tidal cycle. We can now define an effective dilution discharge.

$$Q_{eff} = Q_e + Q + Q_o$$

$$= (Q + Q_e) \left(\frac{S_o}{S_o - S} \right) \qquad (12)$$

This discharge, which is the same as that obtained in equation 10, is the effective discharge available for diluting a waste stream flow.

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As a practical matter the measurement of the cross-sectional average of the tidal cycle average of salinity at a cross-section is difficult and cannot be done with a high degree of accuracy because of the need for an effectively infinite number of measurements to give a true average. The minimum salinity will be observed at the water surface at low slack water; if this value is used equation 12 will yield a minimum dilution discharge.

Suggested regulatory language: It is suggested that section 129.01c(c)(3) be changed to read as follows. Estuaries. The low flow of an estuary shall be the sum of the low flows of all the tributaries entering the estuary, plus the flow of the waste stream, the entire sum being multiplied by the salinity of the ocean or embayment at the mouth of estuary, and divided by the quantity which is the ocean or embayment salinity minus the salinity observed at the water surface above the point of discharge of the waste stream at lower low slack water during a tidal cycle whose lower low is at an elevation equal to or lower than mean lower low water. The salinity above the discharge point to be used in the computation must be observed during a period in which the flow from the tributary streams was no less than that used in the computation.

Example calculation: A river flowing into an estuary has a regulated low flow of 1000 cfs. At the location of an industrial discharge of 100 cfs the salinity observed at the water surface at low slack tide is found to be 19,000 ppm. The estuary discharges into an ocean in which the salinity is 33,000 ppm. The low flow discharge for the estuary is

$$Q_{eff} = (1000 + 100)\frac{33{,}000}{33{,}000 - 19{,}000} = 2596 \text{ cfs}$$

Estuaries with no tributary inflow: Some estuaries or arms of estuaries have no tributary inflow, at least during parts of the year, and there may even be a net negative inflow due to evaporation. For these estuaries a dilution discharge is given by

$$Q_{eff} = Q_e + RP/T \qquad (13)$$

The only prototype measurements of R known to the writer are given by Nelson and Lerseth (1972); they found that at the Golden Gate (the mouth of San Francisco Bay; California) R had a value of 0.17 when the flood tide range was 3.3 ft, and a value of .32 when the tide range was 5.7 feet. The value of R is expected to decrease as one goes landward of the mouth of an estuary. For a given discharge site it is possible to measure a value of R by introducing a stream of florescent dye, such as Rhodamine WT, and observing the build-up of concentration near the discharge point. Unfortunately all dyes known to the writer are subject to decay or adsorption on sediment particles. If one uses the results of a dye study without correcting for loss, the result is a computed dilution discharge greater than would actually exist. Actual rates of dye loss and decay have been measured in rivers; for instance Yotsukura, Fischer and Sayre (1970) found a recovery rate of 77% of the mass of Rhodamine WT injected into the Missouri River near Omaha approximately 40 hours after injection. A similar recovery rate should be applied to computations from dye studies in estuaries. With the proper correction for dye loss it should be possible to measure values of R in estuaries, although the writer is not aware of any experiments in which this has been done. Consequently it is the writer's judgement that a standard for estuaries with no tributary inflow ought to be based on a conservative estimate of R, and that dischargers should be given the option of showing through dye studies that a higher value of R actually exists at their discharge locations. In the writer's judgement a value of R=0.05 would be likely to be exceeded at most estuarine locations, and therefore this value could be selected as a base value.

Recommended regulatory language: In estuaries which have no tributary inflow the low flow shall be defined to be the sum of the waste stream discharge plus the quantity the tidal exchange ratio multiplied by the tidal prism upstream from the point of discharge and divided by the duration of the tidal cycle. The tidal exchange ratio shall be taken to be 0.05 unless shown to have a different value by means of a properly executed dye study.

Example calculation: The tidal prism at the San Mateo Bridge is South San Francisco Bay is approximately 10^{10} cu. ft. (Glenne, 1966). The tide is mixed with a duration of 25 hours. If an industry intends to discharge 100 cfs at this point, the dilution discharge is computed to be:

$$Q_{eff} = 100 + .05 \times \frac{10^{10}}{25 \times 3{,}600} = 5655 \text{ cfs}$$

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This result is in reasonable agreement with the results of a mathematical model of San Francisco Bay, reported by Nelson and Lerseth (1972), which indicated a net circulatory flow at the San Mateo Bridge of approximately 8000 cfs.

Limitation on maximum flow: Near the mouth of an estuary equation 12 may yield unreasonably large dilution discharges, because of division by a number obtained by subtracting two nearly equal quantities. It should be recognized that the computation may not be accurate near the mouth of an estuary, and a limitation on what is considered to be a maximum low flow should be retained. Justification for a maximum dilution flow is given in the next section.

Example of computation of the dilution discharge from a Dye Study: Simmons and Herrmann (1972) describe the results of a dye study carried out in San Diego Bay. Although the dye study was not used to compute dilution discharges or tidal exchange ratios, it can be used as an example of the procedure. In the study 33 cc per min. of a dye with initial concentration of 200,000,000 ppb was released continuously from a point in mid-bay for a period of 15 tidal cycles. The concentration near the injection point at the end of the injection was 8.5 ppb. Assuming no dye decay and that 15 tidal cycles is sufficient to reach equilibrium, the dilution discharge is found to be

$$Q = \frac{33 \text{ cc/min.} \times 2 \times 10^8 \text{ ppb}}{8.5 \text{ ppb}} = 7.76 \text{ cc/min} = 456 \text{ cfs}$$

The tidal prism can be estimated from the map given in the paper to be approximately 24 million cubic meters. Hence the tidal exchange ratio is found to be

$$R = \frac{QT}{P} = \frac{456 \times 25 \times 3600}{24 \times 10^6 = 35.51} = 0.0482$$

Mixing Times in Streams

A number of objectors have questioned EPA's concept of an upper bound on flow (Doudoroff, Fetterolf, Kinney, interalia). I believe that this problem bears additional scrutiny. The proposed effluent standards are based on an assumption of complete mixing between the receiving flow and the waste stream flow. If the waste stream is discharged from a single point or from a series of points occupying only a part of the cross section of the stream some flow distance will be required before complete mixing occurs. In reality complete mixing,

in the sense that the concentration of the effluent is exactly constant everywhere in the receiving flow, requires in theory an infinite flow distance. In practice it is necessary to specify a meaning for "complete mixing"; one definition which easily tested by observation is that the concentration of effluent in the receiving stream does not deviate by more than ± 5% from the mean value. If an effluent is discharged at the side of a stream of rectangular cross section the flow time required to meet this definition is

$$t = 0.4 \frac{B^2}{E} \qquad (14)$$

in which B is the width of the stream and E is the transverse mixing coefficient. If the waste stream is discharged in any other way than at the side of the stream the flow time is less; the exact time can be found mathematically by using the method of images to solve the equation for diffusion across the cross section. The flow distance given by equation 14 is

$$L = UT = 0.4\ UB^2/E \qquad (15)$$

where U is the cross-sectional mean velocity. This is the maximum length of stream for which some parts of the cross section will have a higher concentration of effluent than that given by the "complete mixing" calculation.

To use equations 14 or 15 it is necessary to know the transverse mixing coefficient. A table of values observed in 14 separate experiments has been published by the writer (Fischer 1973), and is attached. Some recent experimental measurements not included in the table have been published by Holley and Abraham (1973). At the present time there is no general agreement among researchers on an exact formula for the transverse mixing coefficient in a natural stream. In the writer's judgement, however, the equation,

$$E = 0.6\ du^* \qquad (16)$$

where d is depth of flow and u^* is shear velocity, represents the best average of experimental observations to date. The writer is not aware of any experimental measurements in natural streams which deviate from equation 16 by more than a factor of 2.

The shear velocity in natural streams depends on the conditions of the banks and bottom, geometry of the stream, and other factors. To use equation 15 for planning purposes it is convenient to use empirical relationships between the shear velocity and the mean flow velocity. A relationship derivable from equations and definitions given in all standard textbooks on hydraulics (see for example Daugherty and Franzini, 1965) is

$$U/u^* = \frac{0.263\ r^{1/6}}{n} \qquad (17)$$

where r is the hydraulic radius, n is Mannings n, and the units are feet and seconds. Chow (1959) gives values of Mannings n for a wide range of stream types, for each of which a photograph of the channel is shown in the book. For natural streams the lowest value of n is 0.025 for a clean, straight, full stage stream with no rifts or deep pools. The normal value for a clean, winding stream with some pools and shoals is 0.040. If a stream has a hydraulic radius of 20' and n = 0.043, the relationship between U and u* is

$$U = 10\ u^* \qquad (18)$$

This equation will be used in the computations below; in the writer's judgement it is a reasonable approximation for most streams, and is likely to be accurate within a factor of 2 except in extremely rough streams.

The proposed rules specify use of a maximum receiving water discharge of 10,000 cfs. One basis for this maximum might be a concern for how much mixing time is required for an effluent to be diluted. The following equations give a means of computing mixing times for a partial discharge. Consider a wide river with a discharge from one side, and an effectively rectangular cross-section (a rectangular cross section is assumed for ease of computation; the same computation could be made for any cross section, but would have to be on a step-by-step basis and would yield results not much different from using a rectangular geometry with the mean depth). Figure 2 shows the effluent plume resulting from the discharge. According to the diffusion equation 95% of the total mass released in the effluent is contained in two standard deviations of diffusing plume. The rate of growth of the plume is given by

$$\sigma^2 = 2Et \qquad (19)$$

Therefore the width of the plume can be estimated by the relations already given to be

$$b = 2\sigma = 2\sqrt{2Et} = 0.69\sqrt{dUt} \qquad (20)$$

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If the receiving water discharge is distributed uniformly over the cross section we can write

$$Q_{eff} = Q\frac{b}{B} = .69\sqrt{d^3 U^3 t} \qquad (21)$$

where Q_{eff} is the discharge which actually participates in diluting the effluent after time t.

Example computation: Leopold and Maddock (1953) give average conditions for the main stems of the Missouri and Mississippi Rivers for a discharge of 100,000 cfs as follows: depth 19 ft., width 1500 ft., velocity 3.5 ft/sec. Suppose that a river has a low flow of 100,000 cfs and the geometry given by Leopold and Maddock. Then the time and distance for complete mixing, as derived above, are

$$t = .4(1500)^2/(.6 \times 19 \times 3.5 \times .1) = 2.25 \times 10^5 \text{ sec}$$
$$L = 3.5 \times 2.25 \times 10^5 = 7.9 \times 10^5 \text{ ft} = 150 \text{ miles}$$

The time and distance required for the edge of an effluent plume to spread such that effluent is mixed into a flow of 10,000 cfs are

$$t = (10,000/.69)^2/(19^3 \times 3.5^3) = 714 \text{ sec}$$
$$L = 2500 \text{ ft}$$

Within this flow distance, however, there is still a large concentration gradient across the flow. The time required for complete mixing with a flow of 10,000 cfs bounded in a flow width of 150 ft (one tenth of the actual channel width) would be

$$t = 2.25 \times 10^3 \text{ sec}$$
$$L = 1.5 \text{ miles}$$

These computations seem to give a reasonable basis on which the Agency might wish to limit the maximum flow used in computations of dilution of an effluent, if the Agency deems it necessary to limit the extent in space and flow time in which concentrations greater than allowable for protection from chronic effects might occur. Because of the approximations used in generating the results, the figures given cannot be considered accurate within less than a factor of 4. A safety factor of this magnitude would be reasonable.

Mixing Time in Estuaries

Mixing times in estuaries are harder to estimate than in streams because of the back-and-forth nature of the flow. Ward and Fischer (1973) report some observations of transverse mixing coefficients of the magnitude of

$$E = 1.0\, d\, u^* \qquad (22)$$

in which u^* is the root mean measure average of the shear velocity caused by the tidal flow. Assuming that estuaries have approximately the same roughness as streams, we can say $U_t = 10\,u^*$, where U is the root mean measure tidal velocity. Then the width of a diffusing plume is given by

$$b = 2\sqrt{2Et} = \sqrt{0.8d\,U_t\,t} \qquad (23)$$

where t is the time that the effluent has been in the receiving water after being discharged. The discharge which participates in the dilution can be derived by an analysis similar to that used for streams. Assume

$$Q_m = \frac{b}{B} Q_{eff} = \frac{b}{B} (Q + Q_e + RP/T) \qquad (24)$$

Usually in tidal flows $Q + Q_e \ll RP/T$, in which we can write to a good approximation.

$$Q_m = \frac{b}{B} R \frac{P}{T} = \frac{b}{B} R\,U_t\,Bd \qquad (25)$$

$$= R\sqrt{0.8\,d^3\,U_t^3}$$

Example calculation: The time required for an effluent to mix with a diluting discharge of 10,000 cfs in the Gironde France, estuary, using the depth of 30 ft. and tidal velocity of 4.5 ft/sec given by Ward and Fischer, and a tidal exchange ratio of 0.05, is

$$10,000 = \sqrt{.05\ 0.8 \times (30)^3 \times (4.6)^3\ t}$$

$$t = 19,000 \text{ sec}$$

$$= 5.3 \text{ hours}$$

Again it should be remembered that this calculation gives the time required for the edge of a plume to reach into a flow of effectively 10,000 cfs. The time required for complete mixing with an effective discharge can be estimated on the basis of the results already given for streams to be on the order of four times as large as the time given above, or approximately one day.

Coastal Concentrations

In their affidavits, Mr. Mancini and Mr. Fetterolf question EPA's quantification of coastal flow. I share their concern. An effluent discharged in a coastal zone usually undergoes dilution in two ways. Firstly, since most effluents are buoyant in ocean water, the effluent plume rises towards the water surface as a buoyant jet. The jet may or may not reach the surface, depending on the state of stratification of the recipient. During the rising jet phase dilution occurs because of entrainment of surrounding water by the jet itself.

Secondly, after the effluent plume has reached the surface or its equilibrium level, and has lost its initial momentum, dilution continues by means of turbulent mixing, as the diluted stream is carried away by the ambient current. Figure 3 shows a sketch of the two processes.

As described in the review paper by Fischer and Brooks (1970), engineers have learned how to predict the dilution in a buoyant jet with reasonable accuracy. The design of diffusers to obtain a specified degree of initial dilution is well understood, and in practice diffusers are used frequently. It is the writer's opinion, on the other hand, that the mechanism of diffusion through turbulent mixing in coastal areas is not sufficiently well understood so that engineers can predict with confidence the degree of dilution to be obtained in an effluent plume through turbulent mixing alone. This conclusion is based on review of published reports of diffusion studies by a number of authors, and a comprehensive review of theory and experiment on oceanic diffusion by Okubo (1970).

It is the writer's opinion, therefore, that the best way to protect against the effects of chronic toxicity is to require that effluents be discharged in such a way that concentrations of toxic materials be reduced to the required concentrations by means of initial dilution alone. An alternative approach, avoiding a diffuser calculation, would be to quantify the low flow discharge through some reasonable and conservative assumptions about the minimum magnitudes of coastal currents and rates of mixing. In the writer's judgement it would be reasonable to adopt the following:

minimum current available for dilution	0.1 ft/sec
minimum vertical spread of the effluent plume	1/4 of the depth
minimum horizontal spread of the effluent plume	10 depths

Using these values gives a dilution current of

$$Q = (0.1) \times (0.25d) \times (10d) = 0.25\ d^2$$

where depth (d) is in feet and Q is in cfs. As an example, this means that a discharge in 50 feet of water would have a dilution discharge of 625 cfs, and a discharge in 100 feet of water would have a dilution discharge of 2500 cfs.

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Suggested regulatory language: Section 129.01c(c)(4) should be changed to read: "The low flow of coastal waters shall be computed by the formula, flow, in cfs, equals 0.25 times the square of the depth of water, in feet, measured at the point of discharge at mean lower low water. An exception to this formula may be granted if the discharger can demonstrate that the effluent is to be discharged in such a way that a larger entrainment flow will be generated by jetting action of the discharge, and if so the low flow shall be taken to be the entrained flow so generated."

Initial Dilution

A number of objectors have questioned EPA's meaning and the logic of specifying "immediate" 1:10 dilution (Duodoroff, Fetterolf, Kinney, inter alia). The objectors are correct in saying that dilution cannot be instantaneous, and that some physically realisable deifnition of "immediate dilution" needs to be given. Initial dilution can be obtained in one of two ways: firstly, by generating an entrained flow through jetting action; secondly, through the mixing action of turbulent motion in the receiving water. It is possible to compute the distance required for mixing by either mechanism to achieve a specified degree of dilution. It is beyond the scope of the writer's field of special knowledge to know what would be an acceptable distance in which to permit attainment of initial dilution; if such a distance were set by others, however, the writer would be able to suggest a computational basis for an effluent standard which would accomplish the necessary dilution within the set distance. For example, a number of experiments (Abraham, 1960) have shown that for a non-buoyant jet into a homogeneous receiving water a dilution of 10 to 1 is achieved in a distance of approximately 55 jet diameters. Mixing due to receiving water flow gives complete mixing over a depth (d) in a downstream distance (L) given by

$$L = 0.4\ U\ d^2/E$$

It is well established (Fischer, 1973) that for vertical mixing

$$E \approx 0.1\ d\ u^*$$

Hence for mixing over a specified partial depth, d', assuming as previously that $U = 10\ u^*$, a downstream distance

$$L = 40\ \frac{(d')^2}{(d)}\ d$$

is required.

Figure 4 gives an illustration of the possible extent of an initial dilution zone if it is determined that immediate diffusion means a dilution within one half the depth of flow ($d' = 0.5\ d$). The zone of initial dilution extends over a width of approximately one depth and a downstream distance of 10 depths. The amount of receiving water flow which participates in the dilution is

$$Q = 1/2\ Ud^2$$

If the zone sketched in Figure 4 is accepted as a reasonable zone for accomplishment of immediate diffusion, than the following language would seem to satisfy the objections raised by the objectors: "Immediate diffusion shall mean that the effluent shall be discharged in such a way that it is diluted with receiving water to the required concentration within a distance from the discharge point of no more than half the depth vertically, and this dilution must occur while the effluent is carried a distance of no more than 10 depths in the stream-wise direction."

It should be noted specifically that adoption of the above definition, in combination with the allowance given in section 129.08c(b)(2) of the proposed rules, should meet the objections of Kodak as contained in the letter from Mr. Tanzer to Dr. Thompson dated March 6, 1974.

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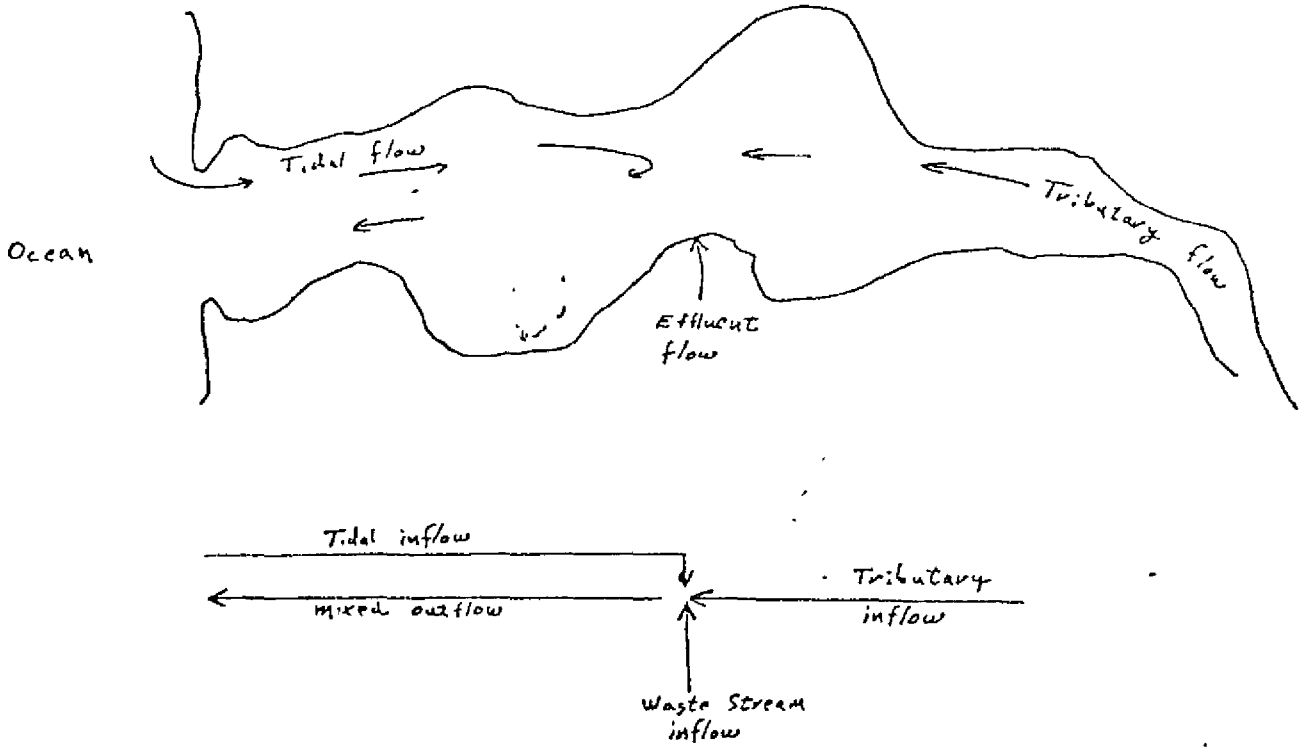

Figure 1. Schematization of estuaries for flow computation.

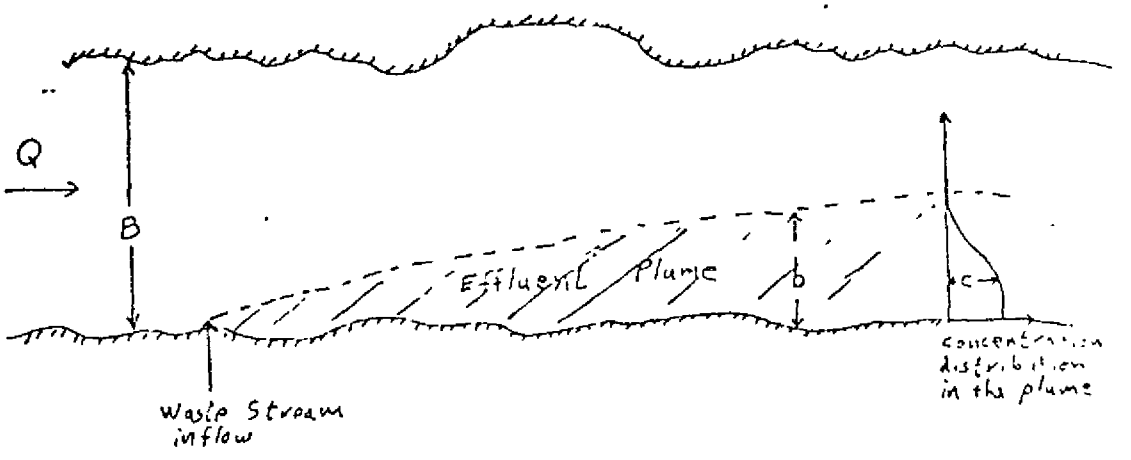

Figure 2. Definition sketch for an effluent plume.

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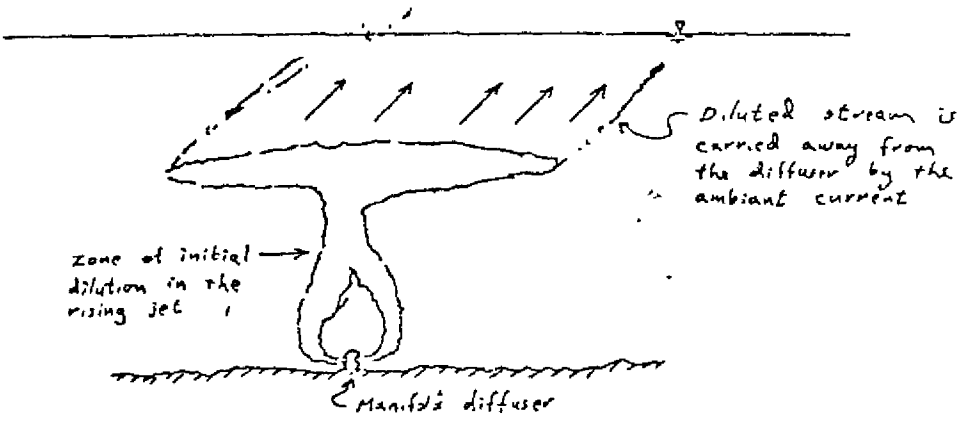

Figure 3. Sketch of an effluent plume from a manifold diffuser in a coastal zone.

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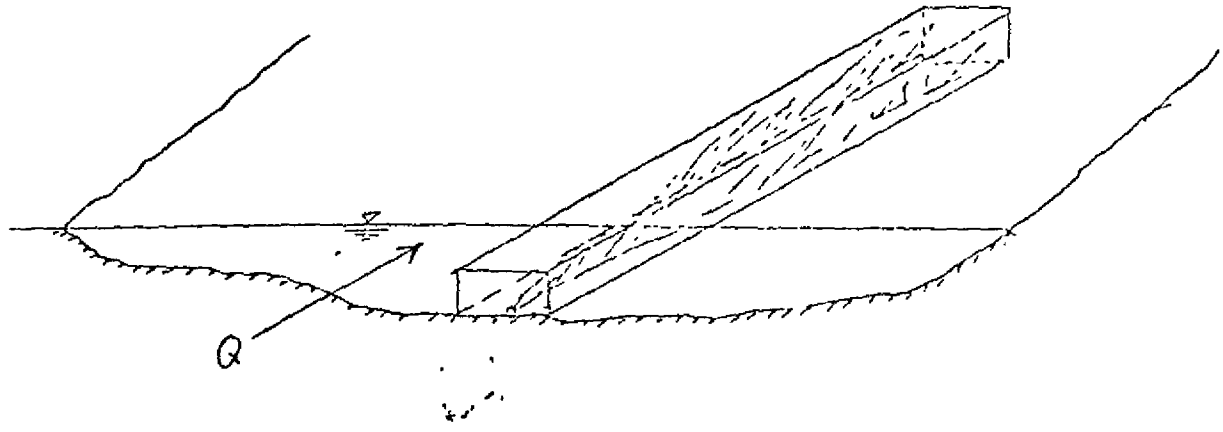

Figure 4. Sketch of a zone for initial dilution.

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Table 1 Experimental measurements of transverse mixing in open channels

	Reference	Channel	Channel width b m	Mean depth of flow d cm	Mean velocity $\bar{u}$ cm/sec	Shear velocity u^* cm/sec	Dispersion coefficient ε_t cm²/sec	$\frac{\varepsilon_t}{du^*}$
Flume & pipe experiments	Orlob (1959)	Lab flume—rough bottom	1.22	1.62–12.8				Average 0.16
	Sayre & Chamberlain (1964)	Lab flume—sand dunes on bottom	2.44	17.4	49.8	3.51	15.0	0.24
	Sayre & Chang (1968)	Lab flume—wooden cleats on bottom	2.38	14.8	23.5	3.81	13.3	0.236
				24.8	34.4	4.90	23.4	0.196
				37.1	47.5	6.01	39.2	0.261
	Engelund (1969)	Lab flume—sand roughness on bottom	2.20	5.5	30.0	3.60	4.0	0.221
				17.3	30.6	1.60	6.5	0.221
	Prych (1970)	Lab flume—13 experiments with rough and smooth bottoms	1.10	3.90–11.1	34.4–14.8	1.96–4.23	1.3–5.73	0.167–0.732 Average 0.304
Laboratory channel experiments	Kalkwijk & Pico (1913)	Smooth tile flume	0.69	15.0	91.5	5.19		0.09
	Elder (1959)	Smooth tile flume	0.36	1.2	21.6	1.39		0.16
	Sayre & Chang (1968)	Lab flume – wooden cleats on bottom	2.38	13.8	23.5	3.81	9.38	0.170
				24.5	31.4	3.90	20.2	0.173
				37.1	37.1	4.01	36.9	0.160
	Sullivan (1968)	Smooth lab flume	0.76	10.2	15.3	0.83	0.90	0.107
				8.95	18.5	0.99	0.97	0.110
				7.33	22.9	1.21	1.18	0.133
	Okoye (1970)	Lab flume, smooth and rough bottoms		See Figure 4 same as above				Average 0.14
	Prych (1970)	Lab flume, smooth and rough bottoms						Average 0.135
Field experiments	Glover (1964)	Columbia River	305	305	155	8.8	1860	0.72
	Fischer (1967b)	Irrigation canal	18.3	69.3	63	6.3	102	0.21
				66.7	66	6.1	102	0.25
	Yotsukura et al (1970)	Missouri River	200	270	175	7.4	1200	0.6

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MAY 3 1974

Statement of Gilman D. Veith

My name is Gilman D. Veith. I am a research chemist at the Environmental Protection Agency National Water Quality Laboratory in Duluth, Minnesota. My duties involve the development of analytical methodology for isolating and identifying persistent organic chemicals in natural waters and aquatic organisms, and research concerning the bioconcentration of organic chemicals in aquatic organisms. I have been employed by the National Water Quality Laboratory since 1972.

I received a B.S. cum laude in chemistry from Augustana College in Sioux Falls, South Dakota, in 1966 and spent the following academic year in graduate studies in chemistry at Cornell University as a Woodrow Wilson Fellow. I completed the M. S. requirements in Water Chemistry at the University of Wisconsin at Madison in 1968 and received a Ph.D. in Water Chemistry from the University of Wisconsin - Madison in 1970.

From 1970 to 1972, I was an Assistant Professor in Water Chemistry at the University of Wisconsin - Madison. My teaching duties included graduate courses in water chemistry, water analysis and advanced techniques in water analysis. The research I have been engaged in during the past six years has centered largely around the occurrence of chlorobiphenyls (PCBs) in the environment. In particular, I have completed studies of the sources of PCBs in selected Wisconsin rivers in the Lake Michigan watershed and the identification and measurement of PCBs in fish from Lake Michigan and Lake Superior.

My testimony will be taken from completed and ongoing research, as well as pertinent results of related studies in the literature. My testimony will

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discuss the analytical limitations associated with PCB measurements and show that PCBs are bioconcentrated in fish by factors greater than 10^5, that the residues resulting by bioconcentration from less than 0.01 ug/l in water have decreased the value of the food resources in Lake Michigan, and that recent evidence confirms the presence of polychlorinated dibenzofurans in PCBs mixtures manufactured in the United States.

ANALYSIS OF CHLOROBIPHENYLS

The identification and measurement of PCBs in environmental samples is inherently difficult because multi-component mixtures are involved. The PCBs include mixtures of hydrophobic chemicals which vary considerably in chlorine content, chemical reactivity, bioconcentration potential, and biological activity. The analytical problems associated with developing methodology for the measurement of complex mixtures are not unique to PCBs but are encountered in numerous other analyses such as chlordane and toxaphene. The need to measure PCBs in large numbers of fish from a bioassay or the Great Lakes where biological variation is subtracted dictates that the methodology permit the necessary numbers of samples to be analyzed, and the interpretation of the residue data is based on more detailed analyses of a small number of representative samples.

A. Isolation of PCBs

The PCBs are generally extracted from water, tissue, or sediment with organic solvents such as hexane, acetonitrile, and acetone. Although the PCB mixtures contain chlorobiphenyls with varying water solubilities (Exhibit 1 summarizes estimates of vapor pressures and solubilities for PCBs and indicates the solubility is a function of composition and varies

by more than 100, the extraction of PCBs in water with immiscible solvents can be designed to assure the extraction is essentially quantitative.

The extraction of large quantities of water (i.e. more than 10 liters) to measure small concentrations of PCBs may require the use of large quantities of solvents which increase the possibility of sample contamination unless high purity reagents are used. To minimize reagent blanks, reverse liquid-liquid partitioning using supports coated with hydrocarbons (Exhibit 2) and adsorption on polymeric foams (Exhibit 3) have been used to extract up to 200 liters of water.

The isolation of the PCBs from the bulk of the co-extractable materials has been based largely on group separations using adsorption differences on florisil, partitioning differences with acetonitrile water systems, and chemical stability to oxidizing acids (Exhibit 4). Although the PCBs are separable from the co-extracted lipids and related materials, the PCBs are more difficult to isolate from interfering chlorinated pesticides, and additional isolation steps are necessary. A frequently encountered interference in PCB measurements is the presence of DDT and its analogs in the sample. One of the more commonly used methods for isolating the PCBs from DDT is silicic acid column chromatograph (Exhibit 5) in which the PCBs are eluted with a non-polar solvent and DDT is eluted with a more polar solvent. However, the adsorption of the many PCBs and DDT analogs may not be restricted to two distinct groups, and incomplete isolation is observed. Exhibit 6 discusses some of the commonly observed problems in using this group separation technique and specifically points out that DDE is often found in the PCB fraction whereas some lesser chlorinated PCBs are eluted in the polar solvent. Thus, the isolation procedure may

alter the composition of the PCB residue, and detailed studies of the separation must be included in the discussion of results.

In addition to the possibility that the PCB residue may be altered, other chemicals or mixtures of chemicals may not be separated from PCBs under conventional methodology. Exhibit 7 illustrates that polychlorinated naphthalenes behave similar to the PCBs on silicic acid. Although analyses using gas chromatography/mass spectrometry (GC/MS) may provide the best estimate of the significance of interferences, other chromatographic techniques such as reverse-phase thin-layer chromatography have been used to complement GLC analyses when GC/MS facilities are not available (Exhibit 8).

B. Identification and Measurement of PCBs

Gas chromatography has served as the basis for PCB measurements due to the capability of separating mixtures and providing quantitative and some qualitative information about the mixtures (Exhibit 9). When the gas chromatograph is interfaced with a mass spectrometer, the mixtures can be resolved and identified with respect to chlorine content and carbon chelation skeleton.

The extent to which the PCB mixtures are resolved in GC/MS analyses is controlled by the GLC conditions. For example, Exhibit 10 illustrates that the PCB mixture, Aroclor 1254, containing 54 percent chlorine can be resolved into at least 69 components. Structures for the components have been proposed; however, since these analyses are prohibited on a routine basis due, in part, to the time required, the chromatograms of the PCB found in residues are often related to those of commercially available PCB mixtures under lower resolution GLC conditions. Under these conditions, the mixtures are relative

into a series of multi-component peaks which provide characteristic chromatograms for most of the commercial mixtures. The composition of these components have been studied (Exhibit 11) and approximately 75 percent of the PCBs in Aroclor 1254 is represented by the six observed peaks having retention times of 70, 84, 98, 104, 125, and 146 relative to DDE=100. The PCB peak at RRT = 70 is comprised of tetrachlorobiphenyls (25 percent) and pentachlorobiphenyls (75 percent). The identification of the components in a residue can be unambiguously identified as the respective PCB components by GC/MS using isotopic abundance ratios as outlined in Exhibit 12.

The quantitation of the PCBs requires numerous analytical considerations. The total PCB content is difficult to measure by integrating total area in the mixture chromatograms because of possible interferences and because the electron capture (EC) response of the EC-GLC analysis varies widely with degree of chlorination and substitution (Exhibits 13 and 14). Webb and McCall (Exhibit 11) have discussed the rationale for the selection of methodology for PCBs. When studying a bioassay system in which a single commercial PCB mixture is added or an effluent which contains principally one PCB mixture, reliable estimates can be made by selecting several interference-free component peaks and comparing sample chromatograms to standard curves prepared from the PCB mixture present.

The same technique has been applied to samples from major watersheds and the oceans where the PCB residues resemble one of the commercially available PCB mixtures. For example, Exhibit 15 summarizes the concentration of PCB mixtures resembling Aroclor 1254 in Lake Michigan fish. It was observed that [illegible] DDE interferences could be removed from the PCBs using the silicic acid technique; however, DDE could not be reproducibly removed.

Therefore, the quantitation of the major components of PCBs were estimated by summing the peak heights of the peaks at 70, 84, 125, 146 and 174 while omitting the doublet at 98 and 104 (relative to DDE = 100) because of the DDE interference. These estimates give a conservative estimate of total PCBs unless attempts to account for other possible mixtures of PCBs present are made.

For samples which contain mixtures of various PCB mixtures, only estimates of the various mixture fractions of the total PCBs present may be possible. The approach to these analytical problems is to use standard curves prepared from mixed PCB mixtures. Exhibit 11 demonstrates the feasibility of dividing complex chromatograms into three major regions corresponding to three PCB mixtures as standards--using selected peaks as the basis of comparison. Exhibit 16 and 17 discuss other methods of using mixed standards and mixtures of isolated components. The majority of monitoring studies reported to date appear to prefer the use of commercially available PCB mixtures as standards, due, in part, to the fact that much of the studies of biological activities are conducted with the commercial mixtures rather than pure isomers.

The perchlorination of all PCB components to decachlorobiphenyl has been used successfully to estimate total PCB concentrations as well as confirm the presence of PCBs. The technique (Exhibits 18 and 19 using $SbCl_5$ or SCl_3AlCl_4 to convert all components of the mixutres to one component greatly enhances the analytical detection limit and, if the average percentage of chlorine of the original mixture can be estimated, the amount of decachlorobiphenyl can be used to calculate the original PCB concentration.

Despite all of the possible errors in the extraction of PCBs and the lack of standard techniques for quantitation, Holden (Exhibit 20) has reported that collaborative studies among 17 laboratories indicated the coefficient of variation for six pesticides and PCBs requiring full processing ranged from ± 10 to ± 18 percent.

BIOCONCENTRATION OF CHLOROBIPHENYLS

An understanding of the bioconcentration of chemicals in organisms is essential to understand the dynamics of the chemicals in the environment. Considerable confusion exists regarding the relative significance of various uptake mechanisms due, in part, to the absence of detailed definition of the systems studied. Much of the early literature assumed that bioconcentration in the aquatic environment was based on the trophic level or food chain concepts.

However, exposures of fish to water containing many hydrophobic chemicals demonstrated that fish can bioconcentrate the chemicals directly from the water. Uptake can be approximated as a first order rate process and that the process appeared to be that of chemical partitioning from the water and the lipids in the tissue. This has led to proposals such as that in Exhibit 21 where the food chain bioconcentration model is rejected in favor of the chemical partitioning model. As with many kinetic models of dynamic systems, multiple pathways are often observed and the relative significance of the various rate expressions is dependent on the conditions imposed. Therefore, the selection of either the food chain or the partitioning model for the aquatic environment requires that the conditions of the ecosystem being considered are defined.

The bioconcentration of PCBs under laboratory conditions in which the mixtures are added only to the water has been studied for a number of aquatic organisms and the observed bioconcentration factor for PCBs in the aquatic environment may differ in different organisms and conditions. For example, Exhibit 21 illustrated that bioconcentration is inversely related to the water solubility (assuming relatively constant lipid solubility). Exhibits 21 and 22 demonstrate that not only does bioconcentration increase with increasing chlorine content, but also that the lipid variations between male and female fish may produce substantial sex-related differences if only the wet weight concentrations of PCBs are considered.

Exhibits 22 and 23 show that the bioconcentration factor of Aroclor 1254 in fathead minnows ranged from 1.1×10^5 to 2.4×10^5 and that the bioconcentration factor for the PCBs to the fathead minnow (where the tissue is generally less than 10 percent lipid) at 25° C ranges from about 0.3×10^5 to 2.5×10^5 with the more highly chlorinated PCBs accumulating to a greater extent. Exhibit 24 shows that Aroclor 1254 is accumulated in pinfish approximately 2×10^4 times that in the water after 35 days of exposure at a salinity of 14 - 34‰. Exhibit 25 suggests that increasing salinity decreases the rate of uptake of lipid-soluble chemicals.

Exhibit 26 shows that the nymph E. danica accumulated Clophen A 50 by an approximate first order process to bioconcentration factors of 3×10^3. Exhibit 27 presents evidence that the bioconcentration factor of Aroclor 1254 in the scud, Gammarus pseudolimnaeus, (was approximately 2.2×10^4), 4.5×10^4 after 48 hours in Daphnia magna, and 1.3×10^4 in mosquito larvae, Culex tarsalis after only 24 hours. After 60 days of exposure to Aroclor 1248 and Aroclor 1248, the maximum bioconcentration factor in Cyprinus was found to

be approximately 3×10^4 and 5×10^4 respectively (Exhibit 28). Exhibit 2[illegible] also reports bioconcentration factors ranging from 2×10^4 to 6×10^4 for bluegills and catfish exposed to Aroclor 1248 and 1254.

Some evidence exists that the bioconcentration factors measured in the laboratory are essentially the same as that found in the river environment. For example, Exhibit 22 shows that the laboratory derived bioconcentration factor for Aroclor 1248 in fathead minnows (~10 percent lipid) ranged from 1×10^5 to 2.4×10^5. Exhibit 29 and 30 report that goldfish in the lower Milwaukee River accumulated Aroclor 1248 approximately 0.7×10^5 to 2×10^5 times (depending on the lipid content) the estimated PCB concentration in the river water. Exhibit 31 presents evidence that the maximum concentration of Aroclor 1254 in Saginaw River shed was 165 ug/gm whereas typical water concentrations averaged 1.1 ug/l to a maximum of 2.9 ug/l. This is equivalent to a bioconcentration factor of 0.6×10^5 to 1.5×10^5, in good agreement with other measurements.

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In summary, for river systems and in laboratory studies many of the aquatic organisms studied appear to bioconcentrate PCB mixtures containing 3, 4, 5, and 6 chlorine atoms per molecule approximately 3×10^3 to 2×10^5 times the concentration in the water they are exposed to.

Occurrence of PCBs in Lake Michigan

The concentration of PCBs in Lake Michigan fish has been studied near Milwaukee in 1969 (Exhibit 9) and throughout the lake in 1971 (Exhibit 5). The data from 1969 indicated that trout and salmon caught near the Wisconsin shore contained from 6 to 25 ug/gm PCBs resembling Aroclor 1254. The more data study presented in Exhibit 15 shows that the PCBs resembling Aroclor 1254 were found at mean concentrations ranging from 2.7 ug/gm in smelt to 15.5 ug/gm in lake trout. The predominant PCB components present in the fish were the [illegible] tri-, tetra-, penta-, hexa-, and heptachlorobiphenyls by GC/MS studies of com-

posite samples.

Exhibit 30 reported concentrations of PCBs similar to Aroclor 1242 in the Milwaukee River, a major tributary to Lake Michigan, up to 2.8 ug/l. Exhibit 32 shows that Aroclor 1242 was found in Illinois tributaries at 0.14 to 1.8 ng/l in 1971 and from 0.01 ug/l to 0.7 ng/l in 1972. Aroclor 1254 was found in the same tributaries at concentrations up to 0.8 ug/l. In an extensive study of PCBs in municipal wastewater effluents in Michigan, the overall mean PCB concentration in all eight-hour composite samples collected was 7.6 ug/l. However, only one of the 50 effluents averaged over 10 ug/l and 44 of the 50 effluents averaged less than 1 ug/l PCB. Exhibit 33 reports similar results for eleven municipal effluents in southeastern Wisconsin in that only one of the eleven effluents contained an average PCB concentration greater than 1 ug/l.

Exhibit 31 provides evidence that the concentrations of PCBs in the intake water of municipalities along the Michigan shore are, with one exception, all below 10 ng/l (ppt). This indicates the minimum bioconcentration factor for smelt in the lake (mean concentration of 2.7 ug/gm) is approximately 3×10^5 and that the minimum bioconcentration factor for the large trout is approximately 15×10^5.

The PCB residues present in the Lake Michigan biota have adversely affected the value of the biological resources of the lake as a food supply for animal feeds. Exhibit 34 is an early indication that Great Lakes fish were suspected of interfering with normal reproduction of mink feeding on fish. Exhibit 35 showed that the effect on mink could not be correlated to the degree of oxidative rancidity or mercury concentration in the fish. Exhibit 36 presents evidence that 5 ug/gm of Aroclor 1254 in the coho salmon diet of mink significantly reduced reproduction and that 1 ug/gm of Aroclor 1254 in the diet caused a slight decrease in reproduction. Exhibit 37 presents similar evidence for mink fed diets enriched with Aroclor 1254. The data indicate that 0.64 ug/gm of

PCB in the rations resulted in only one of 12 mink producing three litters, each of which dies on the first day. Exhibit 15 has shown that the mean concentration of PCBs in coho salmon in Lake Michigan was 11.5 ug/gm in 1971, well above the FDA tolerance level.

Occurrence of PCBs in Lake Superior

Exhibit 38 presents data concerning the mean concentrations of PCBs measured in Lake Superior fish captured in 1972 and 1973 from western Lake Superior. The data indicate that mean PCB concentrations in the smaller, low-lipid content fish contain up to approximately 1.5 ug/gm (on a wet tissue basis) whereas the larger trout contained up to 5.6 ug/gm. Exhibit 39 presents corroborating data for the PCB concentrations in Lake Superior trout obtained by the Michigan Department of Agriculture.

There have been some estimates of PCBs in western Lake Superior water. Exhibit 40 presents estimates of PCBs in the water intake and a bioassay control tank of the National Water Quality Laboratory in Duluth. The estimates were made using the foam plug extract to extract 20 liters of water in triplicate, perchlorinating the extracts with $SbCl_5$, and measuring the decachlorobiphenyl formed. The data indicate that approximately 1-2 ng/l PCBs (ppt as Aroclor 1254) are present in western Lake Superior nearshore water.

The mean concentration of PCBs in the smaller fish is approximately 0.9 ug/gm. This indicates the overall bioconcentration factor in western Lake Superior fish is approximately 9×10^5.

In summary, the data from the Great Lakes demonstrate that PCBs are present in the waters at concentrations below 10 ng/l (ppt), that the concentration of PCBs in the fish are more than 10^5 times that measured in the water, and that the PCB residues which bioconcentrate in fish from water concentrations below 10 ng/l have affected the use of the Lake Michigan resources as a food resource.

Chlorodibenzofurans in Aroclor® PCB Mixtures

Exhibit 41 presents confirmations tetra-, penta- and hexachlorodibenzofurans in the Aroclor® brand of PCBs prepared in the United States. The chlorinated dibenzofuran concentrations decrease with increasing chlorine content of the PCB mixtures. Aroclor 1254 contained eleven chlorodibenzofurans comprised of two terra, four penta and five hexachlorodibenzofurans.

Those exhibits not included in this set are not available because they are either unpublished or the particular reference is not held in the Monsanto Research Center Library. Those exhibits not included are numbers 4, 14, 15, 20, 22, 23, 26-28, 30, 31, 33, 37, 40, 41.

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Westinghouse Electric Corporation Power Systems

Transformer Division

469 Sharpsville Avenue
Sharon Pennsylvania 16146
(412) 981 1611

October 22, 1971

Insulating Fluids Subcommittee
I.E.E.E. Transformer Committee

Gentlemen:

I am enclosing a copy of the paper that I presented at the Electrical Insulation Conference last month which was based on the returns from the industry questionnaire on insulating oil from 143 utilities. A copy of the paper was also sent to all of the utilities that cooperated in the survey.

Since the purpose of the survey was to update the data in the appendix of the Guide for Acceptance and Maintenance of Insulating Oil in Equipment No. 64, I have prepared a new appendix to be added to the next revision of the guide. I am enclosing a copy of the revision for your consideration.

Sincerely yours,

T. K. Sloat

T. K. Sloat, Member
Insulating Fluids Subcommittee

/blh

Enclosure

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12. APPENDIX--RESULTS OF SURVEY OF UNITED STATES AND CANADIAN USERS OF INSULATING OIL.

12.1 General. The initial survey of the United States and Canadian users of insulating oil was made in 1958. The results were published in the original guide. A similar survey has now been made in 1970 and the results contained in this Appendix. A questionnaire was sent to 160 of the larger utilities and 143 replies were used for the summaries reported here. The replies from all laboratories are taken as equal regardless of the number of transformers tested or the size of the utility.

The intent of the resurvey was to show changes in the methods of acceptance and maintenance of insulating oil and these conclusions are noteworthy.

1. From 1958 to 1970 there bas been a significant increase in the number of tests made on each sample of oil.
2. The dielectric breakdown voltage test, D-1816, using VDE electrodes, was adopted in 1960 and is now being used by 17% of the laboratories.
3. Moisture content of insulating oil is being measured by an increasing number of laboratories while the use of the interfacial tension test is decreasing.
4. Cartridge type filters appear to be replacing the older blotter paper filter presses.

The following tables provide information on field and laboratory testing of oils in service to judge its further usefulness, in new equipment as received, and as bulk shipments. The data classifies oils for reconditioning and reclaiming, the methods employed for these processes and the use of inhibitor with treated oil.

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The survey shows that the yearly interval is still most frequently used and the test requirements are more critical as the voltage rating increases. The testing of circuit breaker oil is less critical than transformer oil and the dielectric breakdown voltage tests, D-877, is used as the control.

12.2 Transformer oil - testing of used oil.

Table I shows the average field test and laboratory tests on used oil that permits the continued use of the oil in transformers.

TABLE I

Field and Laboratory Test on Transformer Oil in Service

	Transformer Oil	
Test	Field Test	Laboratory Test
Dielectric breakdown voltage, D-877, KV	25	25
Visual Examination	test made	test made
Neutralization Number, mg. KOH per g. oil	.40	.43
Power Factor, 60 hertz, 25°C percent	1.0	1.0
Color	test made	test made
Dielectric breakdown voltage, D-1816, .04" gap, KV	20	20
Interfacial tension, dynes per cm.	21	--
Moisture content, ppm.	25	31
Pour Point, °F	--	-36
Viscosity, SUS, 100°F	--	62
Flash Point, °F min.	--	275

The importance of the tests are shown by the order in which the various laboratories selected the tests.

Dielectric breakdown voltage, D-877	36%
Neutralization number	23%
Interfacial tension	13%
Power factor	11%
Moisture Content	5%
Color	5%
Visual examination	5%
Dielectric breakdown voltage, D-1816	2%
Pour Point	1%

12.2.1 Classification of service aged transformer oil.

Table II shows the test limits used to classify transformer oil with respect to further usefulness.

Values in parenthesis show the limits established in the 1958 survey.

TABLE II

Classification of Service Aged Transformer Oils

Test	Oils for Continued Service	Oils to be Reconditioned	Oils to be Reclaimed	Oils to be Scraoped
Dielectric breakdown voltage, D-877, KV	24 (23)	23.5 (22)	22	17
Neutralization number, mg. KOH per g. oil	.36(.40)	.40	.40 (.40)	.75 (1.0)
Interfacial tension, dynes per cm.	21 (19)	21	18 (18)	16 (14)
Power factor, 60 hertz, 25°C, percent	1.0 (1.4)	1.2 (1.8)	1.6 (1.4)	3.3
Moisture content, ppm	25	35	60	75

12.2.2 Acceptable limits for Service Aged oils vs. Voltage Class.

Table III shows that the higher the voltage class the more critical the test values.

TABLE III

Acceptable Limits for Service Aged Oil VS Voltage Class

Test	69 KV and below	Above 69 KV thru 288 KV	325 KV and above
Dielectric breakdown voltage, D-877, KV	25	29	31
Neutralization number, mg. KOH per g. oil	.39	.33	.28
Interfacial tension, dynes per cm.	20.5	21.7	30.7
Power factor, 60 hertz, 25°C, percent	1.0	0.7	.30
Moisture content, ppm	30	36	15

12.2.3 Frequency of Testing of Transformers in Service.

Table IV shows that most utilities test transformers on a yearly basis.

TABLE IV

Frequency of Test VS Voltage Class
Percent of Utilities

Frequency of Test	69KV and below	Above 69 KV thru 288 KV	325 KV and above
Every six months	10	13	30
Every year	60	67	53
Every two years	18	14.5	17
Every three years	7.5	3	--
Every four years	3	2.5	--
Every five years	1.5	----	--

12.2.4 Transformer Testing of New Oil.

Table V shows the average test values specified for field and laboratory acceptance of shipments of new oil and in equipment prior to energizing.

TABLE V

Test Values on New Oil as Received and on Oil in Equipment Prior to Energizing

Test	Field Tests on Tank Cars or Tank Trucks	Laboratory Tests on Tank Cars or Tank Trucks	Field Test on New Equip. Before Energizing	Laboratory Test on New Equip. Before Energizing
Dielectric breakdown voltage, D-877, KV	29	29	29	29
Power factor, 60 hertz, 25°C, percent	.22	.35	.34	.21
Neutralization number, mg. KOH per g. oil	.14	.06	.17	.09
Moisture content, ppm	19	30	18	25
Dielectric breakdown voltage, D-1816, .04" gap, KV	23	26	24	25
Interfacial tension, dynes per cm.	25	38	35	36.5
Color	test made	test made	test made	test made
Pour Point, °F	--	-40	--	-30
Flash Point, °F, min.	--	293	--	295
Viscosity, SUS, 100°F	--	63	--	63
Visual examination	test made	test made	test made	test made

12.2.5 Recondition of Transformer Oil.

94.3% of the utilities recondition used oil from transformers. The filter press is the most accepted equipment for this process. However, many users specify a combination of equipment. The following is the equipment most frequently called for.

Filter press	40 percent
Cartridge type filter	33 percent
Vacuum dehydrator	18 percent
Centrifuge	9 percent

Table VI lists the test values of transformer oil before and after reconditioning and shows the values that permits reuse of the oil.

12.2.6 Reclaiming of Oil from Transformers.

54% of the utilities reclaim oil from transformers and 96% use fullers' earth for this purpose. The remaining 4% use activated alumina. Table VI lists the test values of transformer oil prior to reclaiming and the acceptable limits for the oil that permits its reuse. Only 14% of the utilities add inhibitor to reconditioned oil, while 43% add it to reclaimed oil. 0.3% by weight of Ditertiarybutyl para cresol (DBPC) is specified.

TABLE VI

Tests Limits for Transformer Oil Before and After Reconditioning and Reclaiming

Test	Oil requiring Reconditioning*	Oil after Reconditioning	Oil requiring Reclaiming*	Oil after Reclaiming
Dielectric breakdown voltage, D-877, KV	23.5	27	22	28
Neutralization number, mg. KOH per g. oil	.40	.27	.40	.15
Power factor, 60 hertz, 25°C, percent	1.2	.70	1.6	.36
Interfacial tension, dynes per cm.	21	26	18	30
Dielectric breakdown voltage, D-1816, .04" gap, KV	--	23	--	24
Moisture content, ppm	35	23	60	19
Visual examination	test made	--	test made	--

12.3 Circuit Breaker Oil - Testing of Used Oil.

Table VII shows the field tests and laboratory tests on used circuit breaker oil that permit the continued use of the oil.

TABLE VII

Field and Laboratory Tests on Circuit Breaker Oil in Service

	Circuit Breaker Oil	
Test	Field Test	Laboratory Test
Dielectric breakdown voltage, D-877, KV	25	25
Visual examination	test made	test made
Color	test made	test made
Power Factor, 60 hertz, 25°C, percent	1.0	.75
Neutralization number, mg. KOH per g. oil	.45	.40
Dielectric breakdown voltage, D-1816, .04" gap KV	20	21.5
Moisture content, ppm	30	34
Interfacial tension, dynes per cm.	--	21
Pour Point, °F	--	-45
Viscosity, SUS, 100°F	--	62

The importance of the tests are shown by the order in which the various lsboratories selected the tests.

Dielectric breakdown voltage, D-877, KV	47.5
Visual examination	15.7
Power Factor	11.5
Neutralization	9.9
Color	9.1
Interfacial Tension	2.8
Moisture	2.1
Dielectric breakdown voltage, D-1816	.7
Pour Point, °F	.6
Odor	.3

12.3.1 Classification of Service Aged Circuit Breaker Oil.

Table VIII shows the test limits used to classify circuit breaker oil with respect to its further usefulness.

TABLE VIII

Classification of Service Aged Circuit Breaker Oils

Test	Oils for Continued Service	Oils to be Reconditioned	Oils to be Reclaimed	Oils to be Scrapped
Dielectric breakdown voltage, D-877, KV	25 (24)	23 (22)	21 (20)	16 (15)
Neutralization number, mg. KOH per g. oil	.35 (.5)	.35	.42 (.5)	.77 (1.0)
Power factor, 60 hertz, 25°C, percent	1.0 (1.4)	1.2 (1.7)	1.4	2.3
Interfacial tension, dynes per cm	23.6 (18)	26	18.7 (18)	12
Moisture content, ppm	50	35	80	100
Dielectric breakdown voltage, D-1816, .04" gap, KV	18	16.5	--	--

12.3.2 Testing of New Circuit Breakers Oil.

Table IX shows the average field and laboratory test values for acceptance of oil in new circuit breakers.

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TABLE IX

Field and Laboratory Tests Made
on New Circuit Breakers Prior to Energizing

Test	Field Test	Laboratory Test
Dielectric breakdown voltage, D-877, KV	28	29
Power factor, 60 hertz, 25°C - percent	.38	0.10
Visual examination	test made	test made
Neutralization number, mg. KOH per g. oil	.24	.15
Dielectric breakdown voltage, D-1816, .04" gap, KV	21	28
Interfacial tension, dynes per cm.	32	--
Color	test made	test made
Moisture content, ppm	20	30
Viscosity, SUS, 100°F	--	--

12.3.3 Frequency of Testing Circuit Breakers.

The results of the survey showed that 75% of the users test circuit breaker oil every year or sooner. The breakdown is as follows:

Six months	14.2 percent
One year	62.0 percent
Two years	16.4 percent
Over two years	7.4 percent

12.3.4 Reconditioning of Circuit Breaker Oil.

Nearly 96 percent of the users recondition circuit breaker oil. While the filter press is most commonly used for this purpose, many utilities use a combination of equipment. The following is the equipment most frequently specified.

Filter press	47.2 percent
Cartridge type filter	35.7 percent
Centrifuge	8.6 percent
Vacuum dehydrator	8.5 percent

Table X lists the test values of circuit breaker oil before and after reconditioning and shows the values that permit reuse of the oil.

TABLE X

Test Limits for Circuit Breaker Oil
Before and After Reconditioning and Reclaiming

Test	Oil requiring Reconditioning	Oil after Reconditioning	Oil before Reclaiming	Oil after Reclaiming
Dielectric breakdown voltage, D-877, KV	23	27.5	21	27
Power factor, 60 hertz, 25°C, percent	1.2	.80	1.4	.70
Neutralization number, mg. KOH per g. oil	.36	.27	.42	.20
Moisture content, ppm	35	35	80	40
Dielectric breakdown voltage, D-1816, .04" gap, KV	16.5	23	--	21
Interfacial tension, dynes per cm.	26	32	18.7	30

12.3.5 Reclaiming of Circuit Breaker Oil.

Only 27.5 percent of the users reclaim used circuit breaker oil and fuller's earth is used for this purpose. Only 16 percent then add 0.25% DBPC inhibitor to the oil. Table X shows the values for the various tests before and after reclaiming.

12.4 Amount of testing.

The average number of power transformers tested per utility was 775 of all types. The average number of circuit breakers was 830.

Of the users reporting reconditioning, the annual number of gallons per utility was found to be 205,000 gallons. Those reporting reclaiming average 50,000 gallons per year.

Table XI is a breakdown of users based on the gallons of oil reconditioned and reclaimed.

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TABLE XI

Reconditioning and Reclaiming of Oil

Number of Gallons of Oil Treated Annually	Number of Utilities Reconditioning Oil	Number of Utilities Reclaiming Oil
0 - 24,999	21	36
25,000 - 99,999	31	12
100,000 - 999,999	50	12
1,000,000 - and over	7	--

This shows that for every seven gallons of oil that is reconditioned, only one gallon is reclaimed. Economic factors in many cases justify replacing badly used oil with new oil instead of a reclaiming procedure, particularly when the quantities of oil are limited.

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Monsanto

File 1

B2SC P. G. Benignus

April 17, 1975

IEEE Meeting, Orlando, Fla.
April 7-10, 1975

[redacted]
W. B. Papageorge
W. R. Richard - T3A
R. H. Munch - T1B
J. G. Bryant - T1J

TO C. Paton

Summary

1. Al Salazar - ANSI - PCB Steering Committee:

Al offered either NEMA or ANSI headquarters in New York as the most convenient place for the proposed meeting of the ANSI-C107 steering committee to bring members up to date on the status of PCBs relative to EPA.

Shortly Al will send to the electrical industry members of NEMA a copy of EPA's recent announcement that EPA is not only reconsidering the difficulty encountered in obtaining reliable and realistic limits for their list of water pollutants (including PCBs), but moreover EPA is attempting to reassess their over-all objectives, more realistically. Al will send copy to Bill Papageorge, with whom he communicates, frequently.

2. Al Salazar's Comments on Dry Type Transformers:

Makers of dry type transformers are now able to meet ANSI standards for BIL. As the cost of all transformers has increased, during the past 3 years, makers of dry types have been able to construct up to 200KV and 25,000 KVA maximums, (although 10,000 KVA max. seems more realistic). It was said that with "a couple of more" increases in the cost of oil-filled transformers, utilities will switch more to dry types for substations and distribution purposes. While the foregoing was given relative to mineral oil, we can easily insert thoughts relative to the effect of more cost increases for askarel transformers.

3. John Dutton, G.E., Rome, Ga.:

John said that although new orders for Pyranol units are not coming in as strong as they had been, GE still has a good back-log of orders for Pyranol transformers. He does not foresee a suitable fluid replacement for transformer Pyranol.

4. Gary Wilburn, Westinghouse, S. Boston, Va.:

Like GE, Mr. Wilburn indicated that Westinghouse are supplying more askarel transformers than ever before. They are working on their back-log of orders. By the time this runs down, Westinghouse hope that utilities will have received sufficient rate increases to stimulate purchases and that industrial load centers will be in demand, resulting in an upturn in our economy. Otherwise, we will see a let-down for transformer askarel.

Mr. Wilburn is aware that Harry Sheppard objects to having potentially (likely) insoluble material (eg. 4-4 prime isomer) in transformer Inerteen. Accordingly he asked Harry to discuss this with Dr. Munch for better understanding.

5. Dave Shipka, formerly with Hevi-Duty and now with ITE, said that Hevi-Duty are having trouble handling askarel. Also, spoke with Mr. Mutchler of ITE who mentioned what we have known for quite some time, that ITE stopped making askarel units and have their requirements made by Westinghouse.

6. The new IEEE Guide for Transformer Askarel:

The main transformer committee at IEEE has now voted favorable acceptance of the askarel guide prepared by our sub-committee. The ballots along with a few incidental comments are given in the attachment dated April 2, 1975.

7. EPRI - Electric Power Research Institute, Palo Alto, Ca.:

EPRI representatives have been very active at all dielectric technical society meetings held in the recent past. Mr. Herman Halperin and Mr. Norton from EPRI were at the Orlando, Fla. IEEE meeting, where they met privately with people from industry pursuant to arrangements and work contracts supported by at least one half million dollars given EPRI from the utilities who seek guidance on the following:

a) Is it possible to stress and work most electrical apparatus 10 percent harder? This would be the fastest and most direct route to reduce operating costs.

b) Evaluate and provide guidance relative to the transition from naphthenic-based to paraffin-based petroleum oils.

c) Evaluate "synthetic" dielectric fluids as partial or total replacements for petroleum oils used in transformers, circuit breakers, switch gear, etc.

d) Evaluate "synthetic" fluids as replacement for cable oils.

8. EPRI - Cable Oils:

Herman Halperin, 275 Santa Margarita, Menlo Park, Ca. 94025 is the consultant for EPRI in Palo Alto, Ca. who is arranging the work contracts on "synthetic" fluids replacements for cable oils.

He told me that Westinghouse at East Pittsburgh, Pa. has received the contract to test the proposed fluids for cables. This work at Westinghouse will be supervised by their Mr. Steve Mauser, Advanced Systems Technology Group, at East Pittsburgh.

Following fluid evaluation at Westinghouse, the fluid or fluids of most interest will be indicated to General Cable, 800 Rahway Ave., Union, New Jersey where the people under direction of Dr. George Bahder VP will install the fluid in cables and evaluate the results.

I indicated to Mr. Halperin Monsanto's interests in these programs, and mentioned that Monsanto produces dodecylbenzene. As this fluid is already used for cables it is a logical candidate for this program. Additionally it was indicated that Monsanto may wish to submit another fluid.

I have not contacted Mr. Mauser nor Dr. Bahder, pending the wishes of our Marketing and Research people.

P. G. Benignus

cs

[illegible]

[illegible]

[illegible] 1975

Total Ballots sent	87
Total Ballots returned	65 ~~[illegible]~~
Approved	57 ~~56~~ (one with comments)
Disapproved	0
No objections	8 (six with comments)

Comments [illegible] of April 2, 1975

G. E. Sjorring - RTE-ASEA Corporation
We do not recommend use of askarel in transformers.

J. A. Forster - British Columbia Hydro and Power Authority
We have very little experience with askarels because we have minimized their use.

G. H. Hall - Federal Pacific Electric Co.
Do not use askarel.

C. C. Honey - Florida Power and Light Co.
At present we are not involved with transformers filled with askarel raquiring bulk filling.

C. [illegible] - Dept. of Water and Power of Los Angeles
Why does the subcommittee choose to publish an IEEE document rather than update the existing ASTM document D 2283 spec. through Committee D-27?

C. [illegible] - Tennessee Valley Authority
We are concerned that there is not included in this guide, nor a companion guide, information on maintenance and serviceability of service-aged askarels. Also there apparently has been no work done on analysis of dissolved gases in askarel.

James A. Sisson - Federal Pacific Electric Co.
My division does not manufacture askarel-filled transformers.

m

MONS 204626

IEEE GUIDE FOR

ACCEPTANCE AND MAINTENANCE OF

INSULATING OIL IN EQUIPMENT

MONS 204627

ACKNOWLEDGMENT

The Institute wishes to acknowledge its indebtedness to those who have so freely given of their time and knowledge to the preparation of its many publications.

This Guide was prepared by the Subcommittee on Insulating Fluids of the Transformers Committee of the IEEE Power Engineering Society. The membership of this subcommittee was:

E. L. Raab, Chairman

P. G. Benignus
J. E. Dind
J. A. Forster
D. A. Gillies
F. W. Heinrichs
C. Keil
L. A. Kenoyer
R. I. Lowe
A. H. Locke
H. B. Margolis
E. L. Morrison
V. R. Mulhall
R. L. Schwab
T. K. Sloat

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IEEE GUIDE FOR ACCEPTANCE AND MAINTENANCE OF INSULATING OIL IN EQUIPMENT

I. INTRODUCTION

It should be recognized that oil is only one component of an insulation system affecting the overall apparatus characteristics. Adherence to the recommended oil characteristics will assist in obtaining the desired equipment characteristics. Other tests or verification of the integrity of the insulation system are often necessary and required.

Insulating oil contained in equipment such as transformers, circuit-breaking devices, regulators, series and shunt reactors, as received from the manufacturer and as filled at the installation site but prior to service operation, should exhibit certain properties in order to insure satisfactory performance. It is expected that oil contained in equipment as received from the manufacturer when properly sampled from such equipment usually exhibits characteristics slightly different from those obtained from new oil, which has not been in contact with apparatus constructional materials.

Additionally, certain essential properties of insulating oil, used in transformers and circuit-breaking devices, must be retained in service if the oil is to perform its multiple role of electrical-insulating, arc-quenching, and heat-transferring agent reliably. It must have adequate dielectric strength to withstand the electric stresses imposed in service. It must retain a sufficiently low viscosity so that its ability to circulate and transfer heat is not impaired. It must pour readily at low temperatures and have high flash point and fire point for safety. Its dielectric losses should not become excessive. It should not be allowed to become so deteriorated or contaminated that it adversely affects other materials in the apparatus, nor should deterioration products sludge it sufficiently to impair its circulation through cooling ducts.

This Guide attempts to assist the power equipment operator in evaluating oil received in equipment and as filled at the installation site and to assist also in his efforts to maintain his oil in serviceable condition. It recommends and standardizes oil test and evaluation procedures, methods of reconditioning and reclaiming oil when necessary, and routines for restoring oxidation resistance by additions of inhibitors.

II. EVALUATION OF OIL RECEIVED IN NEW EQUIPMENT

Some users of power equipment find it desirable to make field screening tests while others find it more desirable to perform all tests on insulating oil in a laboratory. For those using field screening tests it is quite common to use such tests as dielectric breakdown voltage (most significant), neutralization number, color, and visual condition. Some operators with suitable equipment and trained personnel may wish to gain additional information by use of the interfacial tension test. If water content or power factor results are desired, such tests usually are performed in the laboratory.

In sampling oil contained in apparatus care must be used in order to obtain a representative sample. ASTM Method D923* should be followed.

*ASTM methods referred to in this Guide are obtainable at headquarters of the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa.

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II. EVALUATION OF OIL RECEIVED IN NEW EQUIPMENT - (Cont'd)

Oil exhibiting the following characteristics is considered acceptable:

		ASTM Methods
Dielectric breakdown voltage	26 kV	D877
Neutralization number	0.04 max	D664, D974
Color	1.0 max	D1500
Condition-visual	clear	D1524
Water content - Parts per Million	35 max	D1533
Interfacial tension-dynes/cm (millinewtons/m)	35 min (0.35 min)	D971
Power factor at 25°C	0.001 (0.1%) max	D924
100°C	0.007 (0.7%) max	

III. EVALUATION OF OIL FOR USE IN FILLING APPARATUS AT THE INSTALLATION SITE AND AFTER FILLING SUCH APPARATUS

Manufacturer's instructions for field preparation and oil filling vary because of differences in the design of units and individual preference. Some demand more rigorous quality control. The customer, and the manufacturer should agree on minimum standards.

Shipping Containers - Insulating oil is ordinarily shipped in three types of containers: Oil drums, tank trailers and rail cars, all of which should be clean and dry. Rail cars are usually under control of the refinery and restricted to insulating oil shipment, so that they tend to be cleaner. Highway trailers may be used for transport of many different chemical products as well as insulating oil; therefore, these trailers are more subject to contamination. Because of this, special cleaning and drying procedures are necessary.

Oil drums are the least desirable method of transport but are often used for smaller quantities. Oil drums should be stored either vertically or on their side with the bungs down to prevent moisture entrance. It is a necessity to check the oil in each individual drum or to pump the oil into a single tank for test before processing.

Each tank load or each shipping unit of the oil as received at the customer's site should undergo check test to determine whether the integrity of the oil has been impaired during shipment as the result of handling and storage. The most frequent contaminant is moisture, but the most difficult to detect is chemical.

Sampling procedures should follow recommendation made in ASTM D-923.

Check Tests on Receipt - Oil exhibiting the following characteristics is considered acceptable:

TABLE 1
(On Arrival)

		ASTM Methods
Dielectric Breakdown	30 kV minimum	D877
Dielectric Breakdown	20 kV minimum	D1816 - .040 in. gap
Power factor at 25°C	.05% maximum	D924
at 100°C	0.5% maximum	

III. EVALUATION OF OIL FOR USE IN FILLING APPARATUS AT THE INSTALLATION SITE AND AFTER FILLING SUCH APPARATUS - (Cont'd)

If chemical contamination is expected or is to be guarded against, a 100°C power factor test may be used. This test is very sensitive to chemical contamination. With this test, the limit suggested is 0.5% maximum. Other tests may be performed as desired.

Handling of Oil by Customer and Placing Oil in Storage - Direct transfer from on-site delivery container is preferable. It may be necessary, as a temporary measure, to install the oil in storage tanks. In these instances the following procedures should be used:

Storage tanks should be thoroughly cleaned, wiped dry with dry, clean rags, and flushed with clean oil. Storage tanks should be equipped with breathers and drains situated to effect complete draining. Such tanks are often coated on the inside to prevent rust.

For short-time storage up to 3 months, dehydrating breathers may be installed on the sealed tanks to limit moisture entrance. These breathers should be maintained in a dry condition.

Collapsible or rubber fabric tanks may be used for short-time storage. Care should be taken to insure that they are clean and completely drained of oil residue before filling.

Long-time storage of over 3 months should be avoided, but if such storage becomes necessary, sealed tanks equipped with either dry nitrogen or dry air system may be provided.

Handling and Testing of Oil for Installing in Apparatus - For equipment 345 kV and above, processing is very thorough. The customer and the manufacturer should be in agreement.

Oil dehydrating apparatus should consist of filter driers or vacuum dehydrators of adequate capacity to fulfill the manufacturer's requirements. Degassing may be employed to increase the effectiveness of filling operation if not incorporated in the dehydration apparatus.

In preparation for filling the transformer or apparatus, the tank is evacuated to a value specified by the manufacturer and held for the required time. Then the oil handling system and lines should be completely flushed with dry, treated oil and final quality tests made on the treated oil before introducing any oil into the apparatus.

Suitable connections for filling should then be made to the apparatus. During the filling operation, flow rates should be controlled for the manufacturer's specified flow. In some cases, a maximum of 1500 gallons per hour is specified. The vacuum held on the tank during filling should not exceed the manufacturer's limit.

Apparatus is usually filled from the top at a point well separated from the vacuum opening or as specified by the manufacturer. Conservative practice indicates that the oil should be at a higher temperature than ambient to

III. EVALUATION OF OIL FOR USE IN FILLING APPARATUS AT THE INSTALLATION SITE AND AFTER FILLING SUCH APPARATUS - (Cont'd)

prevent condensation. Some oil treatment apparatus will heat the oil as part of the dehydration process.

The filling process may be varied to suit the capabilities of the equipment employed and the manufacturer's recommendations. In general, throughout the filling operation, the following tests are recommended for monitoring the quality of the oil on transformers 345 kV and above.

Water Content - There are some commercial devices on the market for measuring water content of oil and at least one leased device for continuous monitoring. However, the most common methods in use are based upon the Karl Fischer technique and in particular, ASTM D1533. It is quite common to require moisture contents of 15 ppm or lower for oil admitted to transformers of the higher voltage classes. In fact, some manufacturers require maximum moisture contents of approximately 5 ppm. At these levels, test accuracy is limited at best and is highly dependent upon sampling and testing techniques.

TABLE II
(During Filling)

	345 kV Class	ASTM Methods
Water Content (Karl Fischer Method)	15 ppm max. (10 ppm desirable)	D1533
Dielectric Breakdown	25 kV min.	D1816 - .040" gap
Dielectric Breakdown	(Has only limited usefulness on this class.)	D877
Gas Content	0.5 or below when specified	D831

Dielectric Breakdown - Two methods, ASTM D877 and ASTM D1816 are in current usage and either method may be used to monitor oil processed into transformers. A number of manufacturers believe that ASTM D1816 is more significant for oil used in 345 kV class and higher.

Gas Content - For EHV transformers, it is sometimes desirable to determine dissolved gas content; values of 0.5% and below are commonly specified by manufacturers.

Check Testing of the Oil Before Energizing the Transformer - After the oil filling is completed and after the standing time is completed, tests on the insulating oil should be made before energization.

III. EVALUATION OF OIL FOR USE IN FILLING APPARATUS AT THE INSTALLATION SITE AND AFTER FILLING SUCH APPARATUS - (Cont'd)

TABLE III
(Before Energizing)

	345 kV Class	ASTM Methods*
Dielectric Breakdown	35 kV minimum	D877
Dielectric Breakdown	25 kV minimum - .040" gap	D1816
Power Factor at 25°C	.05 per cent	D924
Neutralization	Acidity .03 maximum	D974
Interfacial Tension	40 dynes minimum	D971
Water Content	15 ppm maximum (10 ppm desirable)	D1533
Gas Content When Specified	0.5 or below or as specified	D831
	115 kV Class through 230 kV Class	
Same as above	Same as above except 30 ppm max. water and 3% max. gas content	Same as above
Gas Content and Water Content, when desired		

NOTE: Reduced BIL's may require processing to meet 345 kV voltage class oil criteria.

	Below 115 kV Class	ASTM Methods*
Dielectric breakdown voltage	26 kV	D877
Neutralization number	0.04 max.	D664, D974
Color	1.0 max.	D1500
Condition - visual	Clear	D1524
Water Content - Parts per Million	35 max.	D1533
Interfacial Tension - dynes/cm (millinewtons/m)	35 min. (0.35 min.)	D971
Power Factor at 25°C	0.001 (0.1%) max.	D924
100°C	0.007 (0.7%) max.	

*ASTM Methods referred to in this Guide are obtainable at headquarters of the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa.

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III. EVALUATION OF OIL FOR USE IN FILLING APPARATUS AT THE INSTALLATION SITE AND AFTER FILLING SUCH APPARATUS - (Cont'd)

Standing before Energization - Most manufacturers suggest, and it is wise to allow oil-filled equipment to stand at least 12 hours after filling and vacuum breaking, and before energization.

Oil circulating pumps should be operated for at least 2 hours of this time. Most manufacturers have specific written procedures for this standing time and pump operation.

IV. CLASSIFICATION OF SERVICE-AGED INSULATING OIL

It is impossible to indicate the value of specific tests and recommended test limits for all possible existing applications of insulating oil in service. It should also be recognized that with the present state of knowledge, no one test can be used as the sole criterion of the condition of oil in service. It is possible, however, to summarize the value and importance of the current tests and to suggest methods of treatment for the oil being examined, such methods being based on current industry experience. Oils in service may be placed in the following classifications based upon the composite evaluation of significant characteristics.

Group I - This group contains oils that are in satisfactory condition for continued use.

Group II - This group contains oils that require only minor reconditioning for further service. (Reconditioning is the mechanical removal of moisture and insoluble contaminants. Typical means employed are filtering and centrifuging.)

Group III - This group contains oils in poor condition. They should be reclaimed or disposed of depending upon economic considerations. (Reclamation involves the use of methods and processes which result in a beneficial chemical change in the oil. Typical means employed are treatments with adsorption agents, acid refining, alkali treatment, etc).

Group IV - This group contains those oils in such poor condition that it is technically advisable to dispose of them.

V. ECONOMIC FACTORS

A review of experience and cost data obtained from many operators and users of transformers reveals a wide variation of practices and cost data involved in the reconditioning and reclaiming of oil. These differences are mostly attributable to the type of system, field inspection operations, and available laboratory and shop facilities. For example, an operator having a large number of pole-type transformers installed over a large area may follow a different practice than one having large transformers in stations in a metropolitan area.

In determining whether or not reconditioning and/or reclaiming or insulating oil is economically justifiable, a number of factors must be considered. Some of these factors are as follows:

V. ECONOMIC FACTORS - (Cont'd)

1. Cost of materials.

2. Disposition of service-aged materials.

3. Total cost of process versus quality of end product.

4. Equipment maintenance and amortization.

5. Cost of collection and storage of oil.

6. Labor and transportation costs.

7. Laboratory costs.

8. Cost of new oil versus cost of reprocessed oil.

9. Loss of oil during reprocessing.

10. Cost of inhibitors and blending process.

11. Value of service-aged oil when used for some other purpose. (One user reports that they utilize certain amounts of this oil for fuel purposes with a value of between 7 and 10 cents per gallon.)

The economic justification of whether reconditioning and/or reclamation work is performed by company personnel or contractors must be resolved on the basis of factors applicable in each particular case..

VI. SAMPLING

Accurate sampling, whether of the complete contents or only parts thereof, is extremely important from the standpoint of evaluation of the quality of the product sampled. Obviously, careless sampling procedures or contamination in the sampling equipment will result in a sample that is not truly representative. This generally leads to erroneous conclusions concerning quality and incurs extra time, effort, and expense involved in securing, transporting, and testing the sample. It is very strongly recommended that all of the procedures and precautions outlined in the latest revision of ASTM D923 (Sampling Electrical Insulating Liquids) be followed.

VII. OIL TESTS AND THEIR SIGNIFICANCE

There are many tests that can be applied to insulating oil for classification purposes. It is recommended that tests be the latest revisions accepted as standards or tentative standards by ASTM. These tests and their significance are as follows:

VII. OIL TESTS AND THEIR SIGNIFICANCE - (Cont'd)

		ASTM Methods
1.	Neutralization Number	D664, D974
	Approximate Acidity Field Test	D1534
	Approximate Acidity and Polar Contamination Spot Test	D1902
2.	Dielectric Breakdown Voltage	D877, D1816
3.	Interfacial Tension	D971
	Interfacial Tension Field Test	D2285
4.	Power Factor	D924
5.	Color	D1500, D1524
6.	Viscosity	D88, D445, D2161
7.	Flash Point	D92
8.	Steam Emulsion	D1935
9.	Specific Gravity (Relative Density)	D1298
10.	Pour Point	D97
11.	Resistivity	D1169
12.	Saponification Number	D94
13.	Gas Content	D831, D1827
14.	Inorganic Chlorides and Sulfates	D878
15.	Corrosive Sulfur	D1275
16.	Sludge	D1313, D2112, D2440
17.	Water in Insulating Oil:	
	Extraction Method	D1315
	Karl Fischer Method	D1533
18.	DBPC Inhibitor in Electrical Insulating Oil	D1473
19.	Visual Examination Field Test	D1524
20.	Sediment and Soluble Sludge in Service-Aged Oil	D1698
21.	Refractive Index and Specific Optical Dispersion	D1807

VII. OIL TESTS AND THEIR SIGNIFICANCE - (Cont'd)

1. Neutralization Number - D664, D974
Approximate Acidity Field Test - D1534
Approximate Acidity and Polar Contamination Spot Test - D1902

The neutralization number for service-aged oils is in general a measure of the acidic constituents of the oil and may be pertinent, if compared to the value for the new product, to detect contamination by substances with which the oil has been in contact, to reveal a tendency toward chemical change or deterioration, or to indicate chemical changes in additives. It may be used as a general guide for determining when an oil should be replaced or reclaimed, provided suitable rejection limits have been established and confirmation is received from other tests.

The tests for field use are not intended to replace standard laboratory tests, but rather are intended as screening tests to minimize the number of field samples submitted to the laboratory. They permit approximate evaluations of the amount of acid and polar constituents and hence of the degree of deterioration and/or contamination of the oil. However, they are only semiquantitative and any decision to replace or reclaim an oil should be confirmed by laboratory tests.

2. Dielectric Breakdown Voltage - D877, D1816

The dielectric breakdown voltage of an insulating liquid is of importance as a measure of its ability to withstand electric stress without failure. It is the voltage at which breakdown occurs between two electrodes under prescribed test conditions. It also serves to indicate the presence of contaminating agents such as water, dirt, or conducting particles in the liquid, one or more of which may be present when low dielectric breakdown values are found by test. However, a high dielectric breakdown voltage does not indicate the absence of all contaminants.

Method D1816 is a recently developed method that prescribes the use of spherically capped electrodes. It is sensitive to small amounts of contaminants and therefore is primarily suitable for use with new oils. Its significance for evaluating service-aged oils is being determined.

Method D877 is generally considered to be more suitable for testing service-aged oils at this time.

3. Interfacial Tension - D971
Field Test - D2285

The interfacial tension between an electrical insulating oil and water is a measure of the molecular attractive force between their unlike molecules at the interface. It is expressed in dynes per centimeter (millinewtons per meter). This test provides a means of detecting soluble polar contaminants and products of deterioration. Soluble-contamination or oil-deterioration products generally decrease the interfacial tension value.

VII. OIL TESTS AND THEIR SIGNIFICANCE - (Cont'd)

4. Power Factor - D924

Power factor is the ratio of the power dissipated in the oil in watts to the product of the effective voltage and current in voltamperes, when tested with a sinusoidal field under prescribed conditions. A high value is an indication of the presence of contaminants or deterioration products such as water, oxidation products, metal soaps, charged colloids, etc.

5. Color - D1500, D1524

The color of an insulating oil is determined by means of transmitted light and is expressed by a numerical value based on comparison with a series of color standards. A rapidly increasing or high color number is an indication of oil deterioration or contamination or both. ASTM D1524 includes a field test suitable for gauging color of service-aged electrical insulating oil.

6. Viscosity - D88, D445, D2161

The viscosity of an oil is its resistance to uniformly continuous flow without turbulence, inertia or other forces. The viscosity of insulating oil is usually measured by the time of flow of a given quantity of oil under controlled conditions. Viscosity is not significantly affected by oil contamination or deterioration but may be useful for identifying certain types of service-aged insulating oils.

7. Flash Point - D92

The flash point of oil is the temperature to which the material must be heated in order to give off sufficient vapor to form a flammable mixture with air under the conditions of the test.

The fire point of oil is the temperature to which the material must be heated in order to cause the oil to ignite and continue burning under the conditions of the test. This temperature is always higher than the flash-point temperature.

A low flash point indicates the presence of hazardous volatile combustible contaminants in the insulating oil. An acceptable flash point is always followed by an acceptable fire point at some higher temperature.

8. Steam Emulsion - D1935

In the steam emulsion test, steam is passed into the oil under prescribed conditions, and the time in seconds required for separation of the oil and the condensed steam is recorded as the steam emulsion number (S. E. Number) of the oil.

The steam emulsion number may be of value in indicating the presence of certain undesirable impurities but has not been widely applied to service-aged oils.

VII. OIL TESTS AND THEIR SIGNIFICANCE - (Cont'd)

9. Specific Gravity (Relative Density) - D1298

The specific gravity (relative density) of an insulating oil is the ratio of the weights of equal volumes of oil and water at 15.56°C (60°F). Specific gravity (relative density) is not significant as such in determining the quality of an oil but may be pertinent in determining suitability for use in specific applications. In certain cold climates, ice may form in equipment exposed to subzero (C) temperatures and may float in oil of high specific gravity (relative density).

10. Pour Point - D97

The temperature at which insulating oil will just flow under the prescribed conditions is known as the pour point.

The pour point has little significance as far as contamination or deterioration is concerned but may be useful for type identification and for determining the type of equipment in which it can be used.

11. Resistivity - D1169

The resistivity (specific resistance) in ohm-centimeters of an oil is the ratio of the direct potential gradient in volts per centimeter paralleling the current flow within the sample to the current density in amperes per square centimeter, at a given instant of time and under prescribed conditions. A low resistivity normally indicates the presence of conductive contaminants, but the test has not been widely applied to service-aged oils.

12. Saponification Number - D94

The saponification number of an oil is the number of milligrams of potassium hydroxide which react with one gram of oil under prescribed conditions. A high saponification number normally indicates the presence of compounds resulting from the oxidation of the oil, added compounds, or contaminants, but the test has not been widely applied to service-aged insulating oils.

13. Gas Content - D831, D1827

The gas content of an insulating oil may be defined as the volume of gas per 100 volumes of oil. In evaluating service-aged oil, this test normally has little significance in determining quality or serviceability of the oil. It will have significance to the operator of apparatus where low gas content is required.

14. Inorganic Chlorides and Sulfates - D878

This test indicates the presence of inorganic chlorides and sulfates by precipitation of insoluble salts by means of the usual analytical procedures. The presence of inorganic chlorides or sulfates is an indication of corrosivity of the oil and of the presence of contaminants.

VII. OIL TESTS AND THEIR SIGNIFICANCE - (Cont'd)

15. Corrosive Sulfur - D1275

This test is designed to detect the presence of free sulfur and combined corrosive sulfur by subjecting copper to contact with oil under prescribed conditions. This test indicates the possibility of corrosion resulting from the presence of either free sulfur or unstable sulfur compounds.

16. Sludge - D1313, D2112, D2440

The sludge value for mineral insulating oil is an indication of its oxidation characteristics. It may be reported as:

A. The percentage of insoluble matter formed in a prescribed length of time, or

B. The amount of acidity formed in a prescribed length of time, or

C. The length of time required for the oil to react with a given volume of oxygen,

when a sample of oil is heated and oxidized under prescribed conditions.

These tests are generally applicable to new and/or reclaimed insulating oils, and D2112 is intended specifically for inhibited oils.

Numerical correlation between results from these laboratory tests and years of service or sludge accumulation in transformers has yet to be established.

17. Water in Insulating Oil:
Extraction Method - D1315
Karl Fischer Method - D1533

Water may be present in insulating oil in several forms. The presence of free water may be disclosed by visual examination in the form of separated droplets or as a cloud dispersed throughout the oil. This type of water invariably results in decreased dielectric strength, which may be restored by filtration or other suitable means. Water in solution cannot be detected visually and is normally determined by either physical or chemical means. Both methods cited are suitable for the determination of water in insulating oil and, depending upon conditions of sample handling and method of analysis, can be used to estimate total-water as well as soluble-water content of oil. The unit of measure of the water is in parts per million. These tests are significant in that they will show the presence of water which may not be evident from electrical tests.

VII. OIL TESTS AND THEIR SIGNIFICANCE - (Cont'd)

18. DBPC Inhibitor in Electrical Insulating Oil - D1473

DBPC (i.e., 2, 6-ditertiary-butyl-para-cresol) is an oxidation inhibitor commonly used in insulating oil to provide added protection against oil oxidation in insulation systems partially or freely exposed to air during service life. Inhibitor effectiveness is a function of base oil type, freedom from contamination, and concentration. Consequently, a method is provided to assay concentration of this inhibitor in insulating oil.

19. Visual Examination Field Test - D1524

This test gauges color and turbidity or cloudiness of an oil, which may indicate the presence of free water or sediment such as metal particles, insoluble sludge, carbon, fibers, dirt, etc. If insoluble contaminants are present, valuable information may be obtained by filtering the oil and identifying the residue.

20. Sediment and Soluble Sludge in Service-Aged Oil - D1698

This test distinguishes between miscellaneous oil-insoluble matter and oil-insoluble sludge and as well signals the presence of oil-soluble sludge. Under certain circumstances, it may be desirable to subject such residues to further laboratory examination and analysis.

21. Refractive Index and Specific Optical Dispersion - D1807

The refractive index of an insulating oil varies with its composition and with the nature and amount of contaminants held in solution. Thus, for service-aged oils, refractive index may be pertinent, if compared to the value for the new product, to detect and estimate any change in composition or degree of contamination.

Specific optical dispersion serves as a quick index for the amount of unsaturated compounds present in an oil: For insulating oils, values above a minimum of about 97 bear a direct relationship to the amount of aromatic compounds present.

These tests have not been widely applied to service-aged oils.

VIII. TESTING PROCEDURES

The procedure followed in applying tests to service-aged insulating oils depends to a great extent on the physical characteristics of the particular property involved and also on the economic considerations. Some users of power equipment find it desirable to make field screening tests while others find it desirable to perform all tests on service-aged insulating oil in a laboratory.

VIII. TESTING PROCEDURES - (Cont'd)

8.1 - Field Screening

Several users of insulating oil use field screening to a great extent. Their experience in this matter indicates that visual condition, dielectric strength, neutralization number, and color will suffice for segregating oil in Groups I and II.

In addition to these tests, some operators with suitable field equipment and trained personnel may wish to gain additional information by use of such tests as interfacial tension and power factor. In interpreting results of these tests consideration must be given to other oil characteristics.

Portable dielectric test sets have been available for some time and have proved quite satisfactory. Several portable test kits are commercially available for testing oil color, acidity, and interfacial tension in the field. As listed and described above, the American Society for Testing and Materials has issued tentative standard field tests for Visual Condition and Color (D1524), for Approximate Acidity and Polar Contamination (D1902), and for Interfacial Tension (D1285).

8.2 - Laboratory Screening

Oils requiring laboratory evaluation will vary widely in condition depending upon the amount of deterioration and the degree of contamination. There are many tests of a research nature that could be applied, but the general use of such tests would be impractical. The following tests, therefore, are believed to be adequate for classifying service-aged oil.

Visual Condition	D1524
Color	D1500
Neutralization Number	D664, D974
Dielectric Breakdown Voltage	D877, D1816
Interfacial Tension	D971
Water	D1315, D1533
Power Factor	D924
Sediment and Oil-Soluble Sludge	D1698
Inhibitor Content	D1698
Viscosity*	D88, D445, D2161
Specific Gravity*	D1298
Pour Point*	D97
Corrosive Sulfur	D1275

*Not essential but can be used to establish type identification.

IX. METHODS OF RECONDITIONING AND RECLAIMING SERVICE-AGED INSULATING OIL

When service-aged insulating oil is to be subjected to reconditioning and/or reclaiming processes it is very strongly recommended that every advantage possible be taken of natural precipitation. Considerable savings can frequently be realized in processing service-aged oil if it is allowed to remain in its container undistrubed for at least 24 hours so that water and suspended solids can settle out. The oil can then be removed without disturbing the residue in the bottom of the container, thus obviating the necessity of removing this residue from the processing machinery.

IX. METHODS OF RECONDITIONING AND RECLAIMING SERVICE-AGED INSULATING OIL

For purposes of this Guide, reconditioning is construed to be the removal of moisture and solid materials by mechanical means while reclaiming is the removal of acidic and colloidal contaminants and products of oxidation by chemical and adsorbent means.

9.1 - Reconditioning - The mechanical means that are used for removing water and solids from oil include several types of filters, centrifuges, and vacuum dehydrators. The extent of the usage of each is shown in the Appendix.

9.1.1 - Filters - Filter devices including the common filter press vary somewhat in form but are based upon the principle of forcing oil under pressure through a series of absorbing materials such as paper. Filters of this type are capable of removing carbon, water, sludge, etc., when they are in suspension but, except for certain special arrangements, they cannot remove them effectively when they are dissolved or in colloidal form. These devices will not remove air and in fact tend to aerate the oil.

The water-removing ability of the filter is dependent upon the dryness of the filter media, and consequently suitable ovens should be provided to dry them before use. When filtering oil containing water, the filter media rapidly come into equilibrium with the water content of the oil and from that time on the filtered oil may contain water up to 100 per cent of saturation at the temperature at which the filtering is done. Since the amount of dissolved water at saturation increases markedly with increasing temperature, filtering at low temperature is more effective in removing water. In the past there has been no satisfactory method of determining when the filter media are too wet for further use. Chief reliance has been placed upon inspection of the media, an increase in input pressure, or a lowering in dielectric strength of the filtered oil. Means are available that permit a continuous indication of the water content of the outgoing oil.

When the oil being processed contains much contamination, it is necessary to change the filter media at frequent intervals. In some cases, the use of a pre-coat applied to the filter medium increases both the efficiency of operation and the length of time that the filter media may be used. Some filters are designed so that small beds of earth or clay may be built up in the press to facilitate the removal of colloids.

9.1.2 - Centrifuges - Another means for separating free and suspended contaminants such as carbon, water, sludge, etc., from oil is the continuous centrifuge. In general, the centrifuge can handle a much greater concentration of contamination than can the conventional filter but cannot remove some of the contaminants as completely as a filter. Consequently the centrifuge is generally found in use for rough bulk cleaning where large amounts of contaminated oil are to be handled. Frequently the output of the centrifuge is put through a filter for the final cleanup. Figure 2 shows such an application.

9.1.2 - Centrifuges - (Cont'd)
The centrifuge cannot remove dissolved water from the oil, and since the centrifuge is sealed with water, the oil leaving the centrifuge may be saturated at the temperature of operation and may contain more dissolved water than when it entered. Neither the centrifuge nor the filter is designed to treat the oil chemically. However, some types of centrifuges have been operated in conjunction with auxiliary tanks containing chemicals such as activated carbon or sodium silicate in order to facilitate the overall results.

9.1.3 - Vacuum Dehydrators - The vacuum dehydrator is an efficient means of reducing the gas and water content of an insulating oil to a very low value. There are two types of vacuum dehydrators in general use today. The guiding principle of both is the same; that is, the oil is exposed to a high vacuum and heat for a short interval of time. In one method the exposure of the oil is accomplished by spraying the oil through a nozzle into a vacuum chamber. In another type of vacuum dehydrator the oil is allowed to flow over a series of baffles inside a vacuum chamber thus forming thin films so that a large surface is exposed to the vacuum. If the oil contains solid matter, it is advisable to pass it through some kind of a filter before processing it in the vacuum dehydrator, since solid contaminants may plug the nozzle of one type of dehydrator or pass through either type without being removed from the oil.

The operation of vacuum dehydrators is continuous. In addition to removing water, vacuum dehydrators will degas the oil and remove the more volatile acids. The other acids, however, will be relatively unaffected and it is doubtful whether the overall acidity of an oil will be much improved by the vacuum dehydration method. In either type of dehydrator some means of automatically recirculating a very wet oil should be provided as a safety device to prevent an excessive water content in the outgoing oil.

9.2 - Reclaiming - The removal of deterioration products is usually accomplished by the use of reclaiming processes involving fuller's earth alone or in combination with certain chemicals. A number of these processes are outlined below.

9.2.1 - Adsorbent Means - Fuller's earth, a naturally occurring clay with a fairly high surface activity, is the material most frequently used for reclaiming oils and is the least expensive. It may be used in its original state, except for drying, crushing, and sizing, or it may be burned, water-washed, steam-treated, or acid-treated. The processed clays are more active but also more expensive. Activated alumina is an efficient adsorbent for impurities found in oil; it is mechanically more stable, and may be reactivated. Oil treated with activated alumina shows a marked rise in steam emulsion number.

In general, reclaiming is done in accordance with one of two methods: a) percolation through coarse clay using either gravity or pressure to force the oil through the clay, or b) contact at an elevated

9.2.1 - Adsorbent Means - (Cont'd)

temperature with finely divided clay. Almost any desired degree of reclaiming may be accomplished by either of the above methods.

9.2.1.1 - Percolation by Pressure - Pressure percolation is similar to gravity percolation in general principle except that the oil is forced through the clay by a pump. Pressure percolators are commercially available and vary in mechanical detail, but all have a chamber to hold a container such as a bag or cartridge filled with clay. The chamber is so designed that oil is admitted around the outside of the clay pack and must pass through a certain amount of clay before leaving the chamber.

These machines are capable of processing large volumes of oil in a relatively short time. They accomplish this by forcing the oil through a comparatively shallow depth of clay at a pressure of about 60 pounds per square inch (400 kilonewtons per square meter) resulting in a short contact time. Since the amount of clay is relatively small with respect to the amount of oil, frequent changes of clay are required.

Specially treated clays are sometimes used for increased efficiency. The output is a graded one for the same reasons that apply to the gravity percolation output, and the same control tests are used.

An advantage of such machines is that they may be brought to the job and used directly on the apparatus whose oil is to be reclaimed. They have been used on energized equipment to a limited extent, and such use will increase when proper safety procedures have been developed.

9.2.1.2 - Percolation by Gravity - Gravity percolation makes use of gravity or the hydrostatic head of a column of oil to force the oil through a column of clay. A typical gravity system, shown schematically in Figure 1 (see page 18), consists of three tanks on different levels. The upper tank is used as a dirty-oil reservoir, the middle tank as the filter containing the fuller's earth, and the lower tank as a blending chamber for the filtered oil. The middle tank is equipped with a strainer-type bottom covered by canvas or filter cloth supporting a 6-foot (2-meter) bed of clay. A float valve controls the flow of oil from the dirty-oil storage tank above so that a constant head of about 15 feet (5 meters) to the filter plate is provided. Once the process is started it continues in operation with very little attention other than periodic sampling. Tests for interfacial tension or acidity are used to control the process.

The output of gravity percolation is a graded one starting with water-white overtreated oil and ending with oil in approximately the same condition as before treatment. To obtain a uniform product, blending is necessary in the third tank in the series. Some convenient mechanical means of agitating the oil in this tank is essential.

IX. METHODS OF RECONDITIONING AND RECLAIMING SERVICE-AGED INSULATING OIL

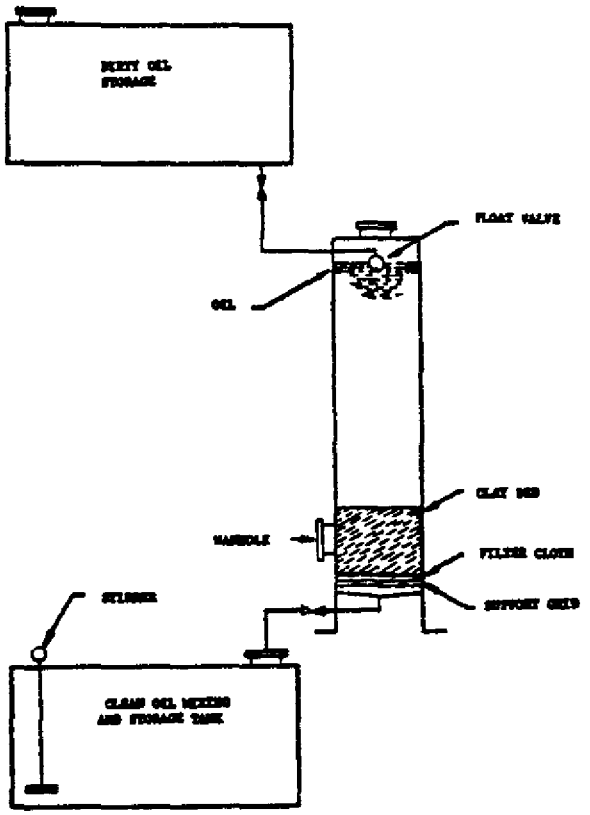

Figure 1. Schematic diagram of gravity-percolation refining apparatus.

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9.2.1.2 - Percolation by Gravity - (Cont'd)

It has been assumed that the undesirable characteristics of the overtreated oil that first comes through the earth are offset in the blending process.

By this method the oil can be treated to any desired degree. The flow rate of such a method is slow, being about 10 gallons per hour per square foot (400 liters per hour per square meter) of filter bed area for an installation such as the one described above. This slow flow rate results in a relatively long contact time which makes for efficient use of the clay.

9.2.1.3 - Contact Process - The contact process for reclaiming oil makes use of 200-mesh (77 mesh per centimeter) fuller's earth and relatively high operating temperatures. This process makes the most efficient use of the earth and produces a uniform product. The degree of reclaiming depends on the amount of earth used which is determined by an analysis of the deteriorated oil.

In a typical commercial apparatus, oil is introduced into a heated mixing chamber as a measured amount of earth is fed in through a hopper. The mixture is stirred as heat is applied, and the process continues until the desired temperature is reached. This operation requires about half an hour. The oil is then dropped into a tank before it is pumped through a filter especially built to accommodate the earth. Much of the oil ordinarily retained in the earth is extracted by the application of compressed air to the filter. A semiautomatic type of apparatus is on the market which recharges itself and operates until the filter becomes filled with clay.

This type of apparatus does not lend itself to the treatment of oil in transformers under load, as, operating on the batch principle, it withdraws oil from the apparatus and does not return the oil for 45 minutes. Also it should not be used to process oil containing DBPC if it is desired to retain the inhibitor in the oil, as this material, when catalyzed by clay, begins to decompose at 100°C.

9.2.2 - Choice of Methods - The choice of reclamation methods that will prove the most practical and economical for a given system depends upon the geographical characteristics of the power system, the existing facilities available for application to such work, and the facts concerning the various types of refining equipment and methods previously described.

For example, on a compact system with large quantities of damaged oil or on a system where oil reconditioning has been done in the past at a central location, the gravity-percolation method of reclaiming has many advantages in requiring a minimum amount of new equipment, attention, and labor. On a system where the oils requiring attention are widely scattered in location, service outages are difficult to

9.2.2 - Choice of Methods - (Cont'd)

obtain, and spare equipment is at a premium, some type of portable pressure percolator may be indicated for reclaiming in the field by recirculating the oil in the equipment. The possibility of recirculation with the equipment alive should be considered in such cases, with due regard to safety measures.

Irrespective of the type of clay treater used, two adjuncts should also be incorporated with the treater.

a) The oil should be put through some device for removing free water before it contacts the clay in order to prevent water from wetting the clay. Water will cause at least partial and possibly complete blocking of the clay, thus making it necessary to discard that batch of clay.

b) The oil coming out of the clay treater should be put through some kind of foolproof, completely automatic dehydrator equipped with positive means and safeguards for preventing any water from being present in the finished product. This is particularly true when recirculating the oil in a transformer and absolutely essential if the reclaiming is done when the transformer is energized.

9.2.3 - Other Reclaiming Methods not Widely Used (See Appendix for extent of usage of each process.)

9.2.3.1 - Reclaiming by Trisodium Phosphate - The trisodium phosphate/activated fuller's earth method (see Figure 2 on page 21) consists in agitating a mixture of oil and trisodium phosphate solution maintained at 80°C for an hour and then allowing the mixture to separate. Most of the spent phosphate solution is drained from the tank, the balance is washed from the oil with a water spray, and the oil is then decanted through a centrifuge and a heater to another tank where 200-mesh (77 mesh per centimeter) activated fuller's earth is added and the mixture agitated. This agitation with clay is maintained for 15 minutes and the clay is allowed to settle out overnight. The oil is again washed with hot water, decanted through a centrifuge, and then dehydrated by passage through a dehydrator or filter.

The process is described as economical and capable of yielding a uniform product by varying the amounts of reclaiming agents as determined by the analysis of the deteriorated oil. The method may be more economical than reclaiming with clay alone for large quantities of badly deteriorated oil.

9.2.3.2 - Reclaiming by Activated Carbon-Sodium Silicate - This method is capable of variation to meet the requirements of different grades of service-aged oil. Under favorable conditions, this process is a continuous one. The method consists of the following basic treatments:

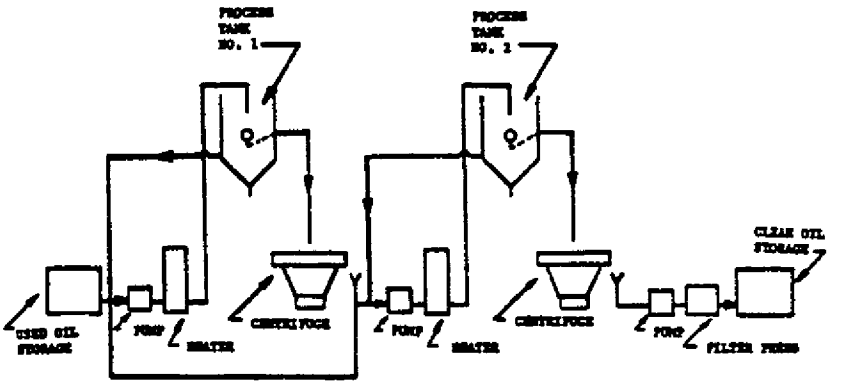

Figure 2. Schematic diagram of trisodium phosphate/fuller's earth refining apparatus.

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9.2.3.2 - Reclaiming by Activated Carbon-Sodium Silicate - (Cont'd)

a) An activated-carbon treatment in which 2 per cent by weight of activated carbon is used.

b) A treatment involving 30 per cent by volume of a 2 per cent sodium silicate solution.

c) A clay treatment in which 2 per cent by weight of activated fuller's earth is used.

The oil is heated to 85°C and is maintained at this temperature until the final filtering operation is reached. The first step consists of a treatment by agitation with activated carbon in cases where the acid rating of the oil is 0.5 milligram of potassium hydroxide per gram or over. This step is necessary to prevent subsequent emulsification of acid oil with the sodium silicate solution. Where the acid value is low, this process may be omitted.

The oil is next decanted through a filter to a second tank where it is paddle-stirred with the sodium silicate solution. It is then run through a centrifuge and the silicate solution discarded.

The oil is now run into a reaction tank with clay added through a hopper. The resulting mixture is agitated, allowed to settle, and then cleaned by passing through a second centrifuge. The oil is then run into a receiving tank and allowed to cool. Finally, it is filtered and run into storage tanks.

When used without activated carbon, the process is continuous with an output of 150 gallons per hour (570 liters per hour). When the activated-carbon treatment is necessary, the process becomes a batch process with an output of 500 gallons per day (1900 liters per day). As both clay and carbon retain about 60 per cent of their weight of oil, the process should be run with a minimum of these materials. Oil in fair condition shows satisfactory characteristics when treated with 1 per cent carbon and clay.

9.2.3.3 - Percolation by Thermo-Siphon Bypass - Good oil which is deteriorating slowly may be held in safe operating condition by applying a special bypass earth filter to the individual transformer. The oil is forced through the filter by the action of the heat generated by the apparatus and the heat dissipated by the filter. As the actuating force is small, the filters must be loosely packed. Coarse activated alumina or 1/4-inch-mesh (0.66 centimeter-mesh) fuller's earth is employed, and the apparatus operates without attention during the active life of the adsorbent.

The bypass method of maintaining oil in usable condition has certain definite limitations. On standby transformers or on circuit breakers it is inoperative as the heat necessary to its operation is not constantly available. The operation of bypass filters might also be termed very "gentle" as little force is available to drive the oil through even a coarse filter bed. There is also the possibility that fine particles of the earth may leak through the system into the processed oil.

X. ADDITION OF INHIBITORS

New insulating oil, as normally refined, contains small amounts of certain chemical compounds that act as oxidation inhibitors. These natural-occurring materials retard oil oxidation until such time as they are consumed. The rate at which the inhibitors in oil are used up is dependent upon such things as the amount of oxygen available, soluble contaminants in the oil, catalytic agents in the oil, and the temperature of the oil. In modern transformers, either sealed to exclude air and moisture or protected by an inert atmosphere, the benefits of the inhibitors can be extended over many years. Once the inhibitor is exhausted, the rate of the oxidation and deterioration of the oil increases. Reclaiming processes, such as acid refining or clay treating, can restore the oil so that it has most of its original characteristics, but this has no effect upon restoring the usefulness of the natural inhibitors occurring in the oil. To overcome this undesirable condition, certain synthetic oxidation inhibitors may be used to extend the life of the reclaimed oil.

A search for suitable oxidation inhibitors has been carried on in many laboratories, and research in this field has been very extensive. These studies have shown not only the benefits of stabilizing reclaimed oil, but also the desirability of fortifying the natural inhibitors in new oil for some applications. Typical types of material which have been studied are sulfides, aromatic amines, phenols, and phosphates. Of the many materials studied, there is one which has been outstanding and has found almost universal approval as an inhibitor. It has been prepared in highly purified form and in sufficient quantities to fill the requirements of the industry. Chemically it is known as 2, 6-ditertiary-butyl-para-cresol, but generally it is referred to as DBPC. Some of the outstanding properties that make this material a desirable inhibitor are:

a) DBPC is a stable and effective oil antioxidant in small concentrations.

b) DBPC is commercially available from at least two sources in a highly purified form.

c) DBPC is readily soluble in oil but insoluble in water.

d) The oxidation products of DBPC are soluble in transformer oil and therefore will not precipitate out as sediment.

e) The oil solutions of the inhibitor are not materially affected by exposure to light.

f) DBPC is insoluble in aqueous alkalis and it is not easily removed from oil by use of the usual reclamation methods of filtration through activated materials if the temperature is maintained below 60°C.

Since in most cases the natural inhibitors are consumed by oil in service, it is important that, following the reclaiming process, an inhibitor such as DBPC should be added to the oil. This can be accomplished either by adding a concentrated solution of the inhibitor to the oil or by the introduction of a dry solid with subsequent heating and agitation of the oil. A concentrated solution

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X. ADDITION OF INHIBITORS - (Cont'd)

of DBPC may be prepared by adding the solid to the oil while gently heating with mechanical agitation. Before the temperature of the oil reaches 50°C, the inhibitor should be in complete solution. Since the solubility of DBPC in insulating oil at usual temperature is about 30 per cent by weight, this figure should not be exceeded. If the solution is to be stored at winter temperatures, a concentrate of 20 per cent by weight should be used for temperatures of 10°C or higher or 10 per cent by weight of temperatures of -30°C to +10°C.

It is undesirable to add more than a small quantity of crystalline material at one time to the oil of an energized transformer. If crystals are added to the oil of an energized transformer, they should be put in slowly and with sufficient time allowed for solution. This can be accelerated by having the oil at a temperature of at least 50°C when the crystals are added. A concentrated solution of an inhibitor should not be added to the oil of an energized transformer since it has appreciably lower breakdown strength than the oil itself. After adding a concentrated solution of an inhibitor to a transformer with the oil at a minimum temperature of 50°C, allow a period of 4 to 6 hours for solution before energizing the transformer. Complete solution is dependent upon the temperature, circulation, and the amount of oil involved. DBPC is stable and soluble in concentrations as high as 20 per cent in insulating oil. However, use of concentrations as high as 20 per cent may give some difficulty due to separation at low temperatures. Solutions of 10 per cent, however, can be handled quite readily. As a general rule, when an inhibitor is added to the reclaimed oil in service, the amount is approximately 0.3 per cent by weight.

XI. SPECIAL CONSIDERATIONS

11.1 - Insulating Oil for Circuit Breakers

11.1.1 - General - The characteristics of insulating oil used in oil circuit breakers, henceforth referred to as circuit-breaker oil, as distinguished from insulating oil for transformers, henceforth referred to as transformer oil, are tabulated below in the table. Most modern circuit breakers require a low viscosity, low pour-point oil since a large percentage of them are used outdoors and in many cases at low temperatures. It should be noted that all circuit breakers are free breathers (open to the atmosphere through a breathing device). This does not prevent the admittance of humid air to the device. There are, however, a number of oil circuit breakers still in use in which it is deemed desirable to use higher viscosity oil. This high-viscosity oil is considered obsolete by most manufacturers and is becoming increasingly difficult to obtain. Although this oil is given the same general tests and treatment as modern oil, caution must be exercised in its usage. Effects which would result from addition of inhibitor, for example, have not as yet been established with certainty. This oil should not be mixed with modern oil except where detrimental effects on cooling effectiveness, freezing temperatures, and other characteristics can be tolerated.

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XI. SPECIAL CONSIDERATIONS - (Cont'd)

11.1.1 - General - (Cont'd)

Type	Color Number	Viscosity 37.8°C (100°C), Seconds	Pour Point °C	Flash Point °C
Modern	1/2 to 1	57	-40	133
Obsolete Outdoor	2 to 3	90	-40	155
Obsolete Indoor	3 to 5	105	-10	185

Although it has been customary to recondition circuit-breaker oil in much the same manner as transformer oil, the problem is somewhat different. Whereas sludging is, or may be, the principal problem in a transformer oil, such is not the case with circuit-breaker oil. Conditions conducive to sludge formation as well as the deleterious effects that are obtained in transformers do not normally apply in the case of circuit breakers. Aside from its dielectric service, the chief function of the transformer oil is to dissipate heat. In a circuit breaker, on the other hand, the chief function of the insulating oil is to quench the arc. In so doing thermal cracking can occur and minute particles of carbon can form. These particles coupled with moisture can lower the dielectric breakdown voltage of the oil. If sufficient quantities of carbon are deposited on the surface of insulating members, a flashover may occur.

11.1.2 - Testing - Testing methods for circuit-breaker oils are the same as those used for transformer oils. Samples are taken in the same manner as for transformer oil at specified intervals, varying from a few months to a year or more. Most users make one or more of the following listed tests on used circuit-breaker oil. Each user includes at least the dielectric breakdown voltage test.

a) Dielectric breakdown voltage;
b) Neutralization number;
c) Interfacial tension;
d) Color;
e) Power factor;
f) Visual examination.

Typical minimum values for oil to be left in service without reconditioning or reclaiming are given in the Appendix.

If it is desired to make additional tests, they should be made in a manner outlined previously for insulating oil. Some users of circuit-breaker oils change or recondition without testing the oil after an agreed-upon number of operations.

XI. SPECIAL CONSIDERATIONS - (Cont'd)

11.1.2 - Testing - (Cont'd)

Viscosity is checked where deemed advisable to verify the type of oil. This characteristic is highly important in determining the speed of operation of circuit breakers at low temperatures.

11.1.3 - Reconditioning - The chief problem in circuit-breaker oil maintenance is to keep it free of water, arcing products, and other contaminants. If visual examination shows the presence of these materials and if the dielectric strength drops below an acceptable value, the oil can be reconditioned provided the acidity, power factor, etc., are good. The accepted method of reconditioning oil is by means of a filter. However, the filter media commonly used are not dense enough to remove all of the colloidal contaminants in the oil. A method being used to some extent to remove colloidal and other contaminants completely is to use a small amount of activated clay with the filter medium. This is accomplished very simply by mixing 1 to 2 cups (one-quarter to one-half liter) of clay with 2-1/2 to 5 gallons (1 to 2 dekaliters) of the oil and then passing it through the filter prior to the main filtering operations. The clay is removed from the oil and deposited on the filter medium thus producing, in effect, a much denser filtering medium. No reduction in acidity of the oil is to be expected since the amount of clay used is extremely small. Typical values to permit reconditioned oil to be put back in service in circuit breakers are given in the Appendix.

In some cases, an oxidation inhibitor is added to the reconditioned oil.

11.1.4 - Reclaiming - In some cases it may be economical to reclaim circuit-breaker oil that has undergone extensive contamination or oxidation. This oil may be reclaimed by one of the processes described previously. To be suitable for circuit-breaker use reclaimed oil must meet certain minimum values. Typical minimum values are given in the Appendix.

XII. APPENDIX

RESULTS OF SURVEY OF UNITED STATES AND CANADIAN USERS OF INSULATING OIL

12.1 - General - The initial survey of the United States and Canadian users of insulating oil was made in 1958. The results were published in the original Guide. A similar survey has now been made in 1970 and the results contained in this Appendix. A questionnaire was sent to 160 of the larger utilities, and 143 replies were used for the summaries reported here. The replies from all laboratories are taken as equal regardless of the number of transformers tested or the size of the utility.

The intent of the re-survey was to show changes in the methods of acceptance and maintenance of insulating oil, and these conclusions are noteworthy.

1. From 1958 to 1970 there has been a significant increase in the number of tests made on each sample of oil.

2. The dielectric breakdown voltage test, D-1816, using VDE electrodes, was adopted in 1960 and is now being used by 17% of the laboratories.

3. Moisture content of insulating oil is being measured by an increasing number of laboratories while the use of the interfacial tension test is decreasing.

4. Cartridge type filters appear to be replacing the older blotter paper filter presses.

The following tables provide information on field and laboratory testing of oil in service to judge its further usefulness, in new equipment as received, and as bulk shipments. The data classifies oils for reconditioning and reclaiming, the methods employed for these processes and the use of inhibitor with treated oil.

The survey shows that the yearly interval is still most frequently used and the test requirements are more critical as the voltage rating increases. The testing of circuit breaker oil is less critical than transformer oil and the dielectric breakdown voltage test, D877, is used as the control.

12.2 - Transformer Oil - Testing of Used Oil

Table 1 shows the average field tests and laboratory tests on used oil that permits the continued use of the oil in transformers.

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XII. APPENDIX - (Cont'd)

TABLE I

AVERAGE LIMITS OF ACCEPTABILITY FOR TRANSFORMER OIL IN SERVICE

	Transformer Oil	
Test	Field Test	Laboratory Test
Dielectric Breakdown Voltage D877, kV	25	25
Visual Examination	Test made	Test made
Neutralization Number, mg KOH per g. oil	.40	.43
Power Factor, 60 Hertz, 25°C per cent	1.0	1.0
Color	Test made	Test made
Dielectric Breakdown Voltage D1816, .04" gap, kV	20	20
Interfacial tension, dynes per cm	21	-
Moisture content, PPM	25	31
Pour Point, °F	-	-36
Viscosity, SUS, 100°F	-	62
Flash Point, °F Min.	-	275

The importance of the tests is shown by the order in which the various laboratories selected the tests.

Dielectric breakdown voltage, D877	36%
Neutralization number	23%
Interfacial tension	13%
Power factor	11%
Moisture content	5%
Color	5%
Visual examination	5%
Dielectric breakdown voltage, D1816	2%
Pour Point	1%

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XII. APPENDIX - (Cont'd)

12.2.1 - Classification of Service-Aged Transformer Oil

Table II shows the test limits used to classify transformer oil with respect to further usefulness.

TABLE II

CLASSIFICATION OF SERVICE-AGED TRANSFORMER OILS
(Values in parentheses show the limits established in the 1958 survey.)

Test	Oils for Continued Service		Oils to be Reconditioned		Oils to be Reclaimed		Oils to be Scrapped	
Dielectric breakdown voltage, D877, kV	24	(23)	23.5	(22)	22		17	
Neutralization number, mg KOH per g. oil	.36	(.40)	.40		.40	(.40)	.75	(1.0)
Interfacial tension, dynes per cm	21	(19)	21		18	(18)	16	(14)
Power factor, 60 Hz, 25°C, per cent	1.0	(1.4)	1.2	(1.8)	1.6	(1.4)	3.3	
Moisture content, PPM	25		35		60		75	

12.2.2 - Acceptable Limits for Service-Aged Oils versus Voltage Class

Table III shows that the higher the voltage class the more critical the test values.

TABLE III

Acceptable Limits for Service-Aged Oil versus Voltage Class

Test	69 kV and Below	Above 69 kV thru 288 kV	325 kV and Above
Dielectric breakdown voltage, D877, kV	25	29	31
Neutralization number, mg KOH per g oil	.39	.33	.28
Interfacial tension, dynes per cm	20.5	21.7	30.7
Power factor, 60 Hertz, 25°C, per cent	1.0	0.7	.30
Moisture content, PPM	30	36	15

XII. APPENDIX - (Cont'd)

12.2.3 - Frequency of Testing of Oil in Transformers in Service

Table IV shows that most utilities test transformers on a yearly basis.

TABLE IV

Frequency of Test versus Voltage Class
Per cent of Utilities

Frequency of Test	69 kV and Below	Above 69 kV thru 288 kV	325 kV and Above
Every six months	10	13	30
Every year	60	67	53
Every two years	18	14.5	17
Every three years	7.5	3	-
Every four years	3	2.5	-
Every five years	1.5	-	-

12.2.4 - Testing of New Oil for or in Transformers

Table V shows the average test values specified for field and laboratory acceptance of shipments of new oil and in equipment prior to energizing.

TABLE V

Test Values on New Oil as Received
and on Oil in Equipment Prior to Energizing

Test	Field Tests on Tank Cars or Tank Trucks	Laboratory Tests on Tank Cars or Tank Trucks	Field Test on New Equip. Before Energizing	Laboratory Test on New Equip. Before Energizing
Dielectric breakdown voltage, D877, kV	29	29	29	29
Power factor, 60 Hz, 25°C, per cent	.22	.35	.34	.21
Neutralization number, mg KOH per g oil	.14	.06	.17	.09
Moisture content, PPM	19	30	18	25
Dielectric breakdown voltage, D1816, .04" gap, kV	23	26	24	25
Interfacial tension, dynes per cm	25	38	35	36.5
Color	Test made	Test made	Test made	Test made
Pour Point, °F	-	-40	-	-30
Flash Point, °F, Min.	-	293	-	295
Viscosity, SUS, 100°F	-	63	-	63
Visual examination	Test made	Test made	Test made	Test made

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XII. APPENDIX - (Cont'd)

12.2.5 - Reconditioning of Transformer Oil

94.3% of the utilities recondition used oil from transformers. The filter press is the most accepted equipment for this process. However, many users specify a combination of equipment. The following is the equipment most frequently called for:

Filter press	40 per cent
Cartridge-type filter	33 per cent
Vacuum dehydrator	18 per cent
Centrifuge	9 per cent

Table VI lists the test values of transformer oil before and after reconditioning and shows the values that permit reuse of the oil.

12.2.6 - Reclaiming of Oil from Transformers

54% of the utilities reclaim oil from transformers and 96% use fuller's earth for this purpose. The remaining 4% use activated alumina. Table VI lists the test values of transformer oil prior to reclaiming and the acceptable limits for the oil that permit its reuse. Only 14% of the utilities add inhibitor to reconditioned oil, while 43% add it to reclaimed oil. 0.3% by weight of ditertiary-butyl para-cresol (DBPC) is specified.

TABLE VI

Test Limits for Transformer Oil before and After Reconditioning and Reclaiming

Test	Oil Requiring Reconditioning*	Oil After Reconditioning	Oil Requiring Reclaiming*	Oil After Reclaiming
Dielectric breakdown voltage, D877, kV	23.5	27	22	28
Neutralization number, mg KOH per g oil	.40	.27	.40	.15
Power factor, 60 Hz, 25°C, per cent	1.2	.70	1.6	.36
Interfacial tension, dynes per cm.	21	26	18	30
Dielectric breakdown voltage, D1816, .04" gap, kV	-	23	-	24
Moisture content, PPM	35	23	60	19
Visual examination	Test made	-	Test made	-

*From Table II.

XII. APPENDIX - (Cont'd)

12.3 - Circuit Breaker Oil - Testing of Used Oil

Table VII shows the field tests and laboratory tests on used circuit breaker oil that permit the continued use of the oil.

TABLE VII

Field and Laboratory Tests on Circuit Breaker Oil in Service

Test	Circuit Breaker Oil	
	Field Test	Laboratory Test
Dielectric breakdown voltage, D877, kV	25	25
Visual examination	Test made	Test made
Color	Test made	Test made
Power factor, 60 Hertz, 25°C, per cent	1.0	.75
Neutralization number, mg KOH per g. oil	.45	.40
Dielectric breakdown voltage, D1816, .04" gap kV	20	21.5
Moisture content, PPM	30	34
Interfacial tension, dynes per cm.	-	21
Pour Point, °F	-	-45
Viscosity, SUS, 100°F	-	62

The importance of the tests is shown by the order in which the various laboratories selected the tests.

Dielectric breakdown voltage, D877, kV	47.5
Visual examination	15.7
Power factor	11.5
Neutralization	9.9
Color	9.1
Interfacial tension	2.8
Moisture	2.1
Dielectric breakdown voltage, D1816	.7
Pour Point, °F	.6
Odor	.3

MONS 204662

XII. APPENDIX - (Cont'd)

12.3.1 - Classification of Service-Aged Circuit Breaker Oil

Table VIII shows the test limits used to classify circuit breaker oil with respect to its further usefulness.

TABLE VIII

Classification of Service-Aged Circuit Breaker Oils
(Values in parentheses show the limits established in the 1958 survey.)

Test	Oils for Continued Service		Oils to be Reconditioned		Oils to be Reclaimed		Oils to be Scrapped	
Dielectric breakdown voltage, D877, kV	25	(24)	23	(22)	21	(20)	16	(15)
Neutralization number mg. KOH per g. oil	.35	(.5)	.35		.42	(.5)	.77	(1.0)
Power factor, 60 Hz., 25°C, per cent	1.0	(1.4)	1.2	(1.7)	1.4		2.3	
Interfacial tension, dynes per cm	23.6	(18)	26		18.7	(18)	12	-
Moisture content, PPM	50		35		80		100	
Dielectric breakdown voltage, D1816, .04" gap, kV	18		16.5		-		-	

12.3.2 - Testing of New Circuit Breaker Oil

Table IX shows the average field and laboratory test values for acceptance of oil in new circuit breakers.

TABLE IX

Field and Laboratory Tests Made
on New Circuit Breakers Prior to Energizing

Test	Field Test	Laboratory Test
Dielectric breakdown voltage, D877, kV	28	29
Power factor, 60 Hertz, 25°C, per cent	.38	0.10
Visual examination	Test made	Test made
Neutralization number, mg. KOH per g. oil	.24	.15
Dielectric breakdown voltage, D1816, .04" gap, kV	21	28
Interfacial tension, dynes per cm.	32	-
Color	Test made	Test made
Moisture content, ppm	20	30
Viscosity, SUS, 100°F	-	-

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XII. APPENDIX - (Cont'd)

12.3.3 - Frequency of Testing Circuit Breaker Oil

The results of the survey showed that 75% of the users test circuit breaker oil every year or sooner. The breakdown is as follows:

Six months	14.2 per cent
One year	62.0 per cent
Two years	16.4 per cent
Over two years	7.4 per cent

12.3.4 - Reconditioning of Circuit Breaker Oil

Nearly 96 per cent of the users recondition circuit breaker oil. While the filter press is most commonly used for this purpose, many utilities use a combination of equipment. The following is the equipment most frequently specified.

Filter press	47.2 per cent
Cartridge-type filter	35.7 per cent
Centrifuge	8.6 per cent
Vacuum dehydrator	8.5 per cent

Table X lists the test values of circuit breaker oil before and after reconditioning and shows the values that permit reuse of the oil.

TABLE X

Test Limits for Circuit Breaker Oil
Before and After Reconditioning and Reclaiming

Test	Oil Requiring Reconditioning	Oil After Reconditioning	Oil Before Reclaiming	Oil After Reclaiming
Dielectric breakdown voltage, D877, kV	23	27.5	21	27
Power factor, 60 Hertz, 25°C, per cent	1.2	.80	1.4	.70
Neutralization number, mg KOH per g. oil	.36	.27	.42	.20
Moisture content, PPM	35	35	80	40
Dielectric breakdown voltage, D1816, .04" gap, kV	16.5	23	-	21
Interfacial tension, dynes per cm.	26	32	18.7	30

12.3.5 - Reclaiming of Circuit Breaker Oil

Only 27.5% of the users reclaim used circuit breaker oil and fuller's earth is used for this purpose. Only 16% then add 0.25% DBPC inhibitor to the oil. Table X shows the values for the various tests before and after reclaiming.

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XII. APPENDIX - (Cont'd)

12.4 - Amount of Testing

The average number of power transformers tested per utility was 775 of all types. The average number of circuit breakers was 830.

Of the users reporting reconditioning, the annual number of gallons per utility was found to be 205,000 gallons. Those reporting reclaiming average 50,000 gallons per year.

Table XI is a breakdown of users based on the gallons of oil reconditioned and reclaimed.

TABLE XI

Reconditioning and Reclaiming of Oil

Number of Gallons of Oil Treated Annually	Number of Utilities Reconditioning Oil	Number of Utilities Reclaiming Oil
0 - 24,999	21	36
25,000 - 99,999	31	12
100,000 - 999,999	50	12
1,000,000 - and over	7	-

This shows that for every seven gallons of oil that is reconditioned, only one gallon is reclaimed. Economic factors in many cases justify replacing badly used oil with new oil instead of a reclaiming procedure, particularly when the quantities of oil are limited.

MONS 204665

CANADIAN GENERAL ELECTRIC COMPANY LIMITED

INDUSTRIAL APPARATUS DEPARTMENT

107 PARK STREET NORTH PETERBOROUGH ONTARIO · TELEPHONE 742-7711

26 April 1972

Members
Insulating Fluids Subcommittee
IEEE Transformers Committee

During our meeting in St. Louis I was requested to submit information to the members on the "Dornenburg Method" that was introduced by John Dind.

Attached is a copy of pages 15 to 20 inclusive of CIGRE paper 15-07 of the 1970 session, which is in fact Dornenburg's report to that meeting. The graph that John distributed at the meeting is presumably a copy of Hydro Quebec's working graph for application of this method of interpretation.

Although we have not had the need or opportunity to conduct physical and visual observations of internal core and coil structures of transformers taken from service that have presented themselves to John and his people, I can indicate that in our limited experience we have not found any significant contradiction to the Dornenburg interpretation during the last year.

One question that arose at the St. Louis meeting was how did you use this graph if no acetylene was detected. It must be remembered that this procedure is intended only as an aid in interpreting problems when gas <u>is</u> being generated in significant quantities. If there are no significant quantities of gases, there is no need for interpretation.

I suggest you see how it fits your experience.

Yours truly,

V. R. Mulhall
Insulating Fluids Subcommittee

/jt
ENCL.

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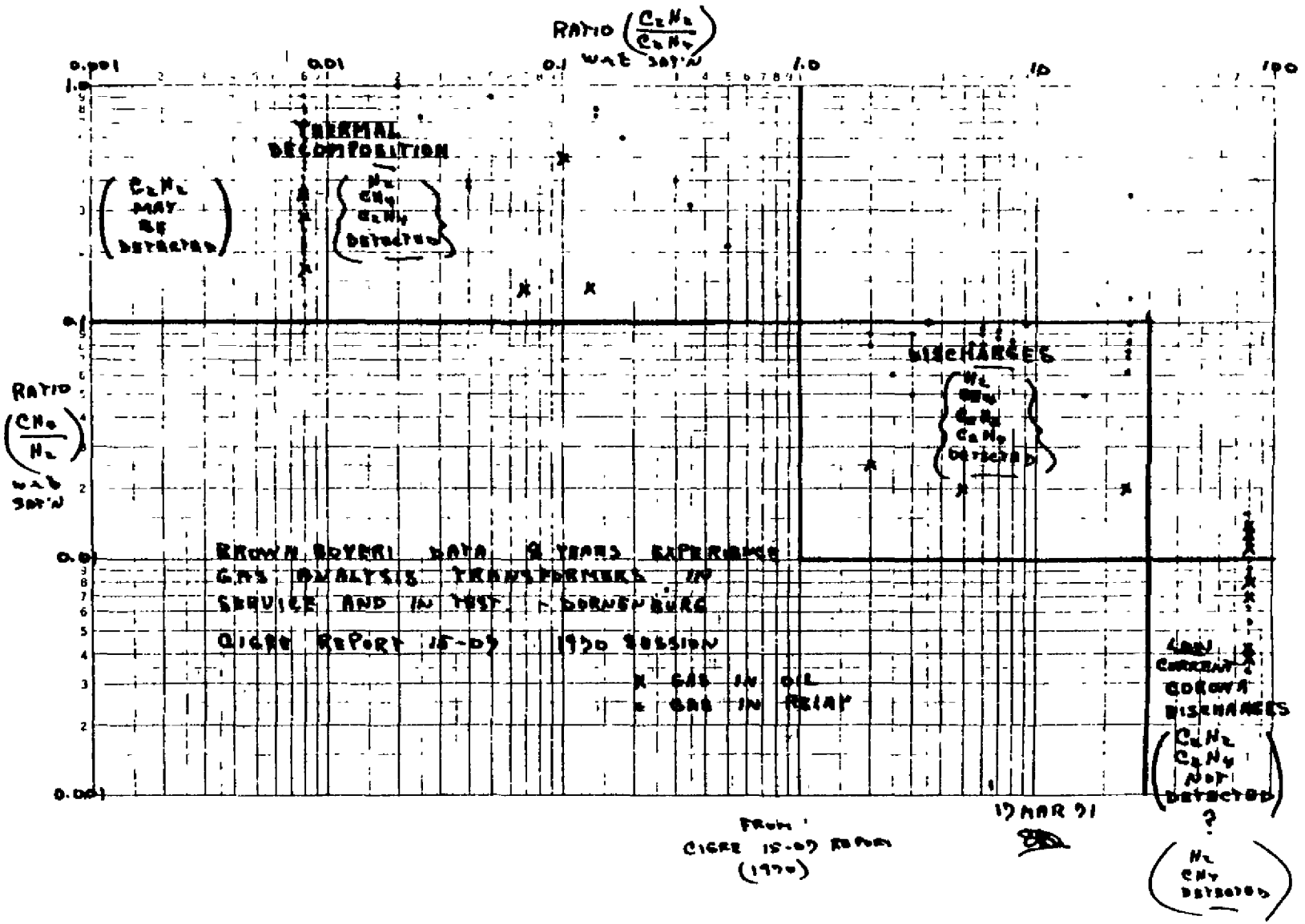
RATIO $\left(\frac{C_2H_2}{C_2H_6}\right)$
wet soil
0.001
0.01
0.1
1.0
10
100
THERMAL DECOMPOSITION
C_2H_2 may be detected
H_2 CH_4 C_2H_4 detected
DISCHARGES
H_2 CH_4 C_2H_4 C_2H_2 detected
RATIO $\left(\frac{CH_4}{H_2}\right)$
wet soil
BROWN BOVERI DATA 8 YEARS EXPERIENCE
GAS ANALYSIS TRANSFORMERS IN
SERVICE AND IN TEST - DORNENBURG
CIGRE REPORT 15-05 1970 SESSION
X GAS IN OIL
+ GAS IN AIR
FROM CIGRE 15-05 REPORT (1970)
17 MAR 71
LOW CURRENT CORONA DISCHARGES
C_2H_2 C_2H_6 Not Detected
?
H_2 CH_4 Detected

MONS 204667

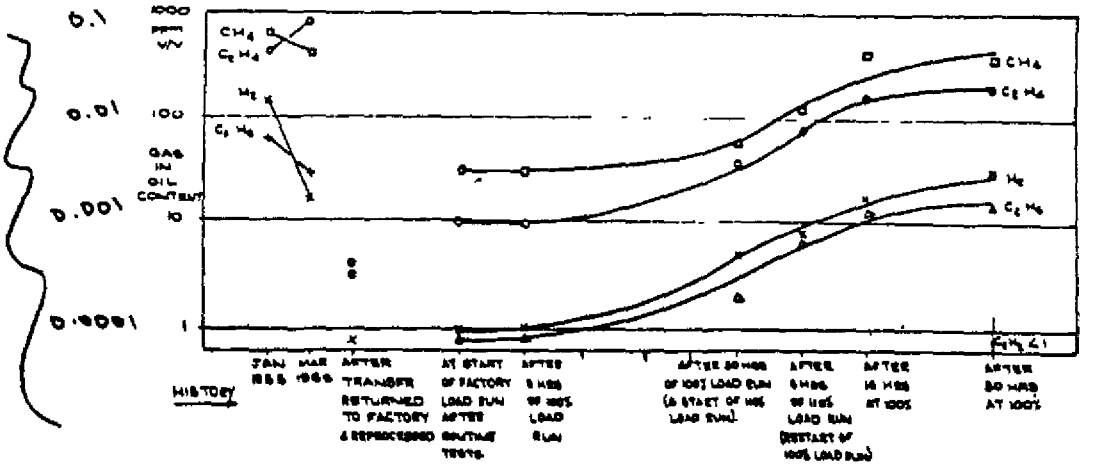

Figure 7 - 310 MVA 275 kV generator transformer

bons. It is also necessary to study the rate of loss of gas from a transformer, for the gases which are at present being measured are those which remain in the oil and not necesaarily those which are formed. The sampling technique also requires attention. Work on all these aspects is in hand in the C.E.G.B. and it is hoped to report on it fully on a future occasion.

6. SECOND OPERATING EXPERIENCE ON TRANSFORMERS

1. Statement of the problem - If faults occur in a transformer so that discharges are caused or there is local overheating, then gaseous decomposition products (hydrogen, hydrocarbons) are always formed from the oil which surrounds all active parts of the transformer. If solid insulation is also affected, then oxides of carbon are produced (carbon monoxide, carbon dioxide). The gases produced in this manner rise in the oil ; then they are either completely dissolved or a part is dissolved in the oil and the remainder reaches, in the case of the type of transformer under consideration, the gas collector relay. Gas which collects there is thus only a part of that which was originally produced, and it contains also air, which was previously dissolved in the oil and was flushed out by exchange with the gas bubbles. Mainly due to the differences in solubility of the gas components the gas which is collected has a different composition from that which was produced at the position of the fault.

All this is known and often the question has been asked whether the analysis of the gases collected can make any contribution at all to the diagnosis of faults.

Below a report is given on experience of Brown Boveri & Co. on the formation of gases in transformers, data having been collected in the last eight years. This is intended to contribute to the discussion of whether gas analyses can give any help in fault diagnosis. The results given below are restricted to transformers with gas collector relays and conservator, as well as to mineral oils as liquid insulation. The gas formation occurred partly in operation, and partly during tests ; the decomposition products which were observed were produced by the decomposition of organic insulating materials, i.e. from the mixture of hydrocarbons constituting the insulating oil, and the solid insulation, which was mainly paper and transformer board.

2. Results of the investigations of decomposition gases produced under operating conditions

2.1. Gas formation in unfaulted operating transformers - So far it has not been possible to detect any decomposition products of the insulating oil during unfaulted operation. On the other hand, when transformers are on load it is noted that carbon monoxide is produced inside the transformer. The production of gas is very small and can be recognised only by the fact that the amount of carbon monoxide dissolved in the oil slowly increases. The phenomenon indicated that the paper is, to a small extent, subjected to thermal attack.

Figure 8 shows the increase in the concentration of dissolved carbon monoxide[1], which was measured on several transformers of the same type at approximately the same loading. In a reserve transformer which was not in operation there was, on the other hand, no change in the content of carbon monoxide.

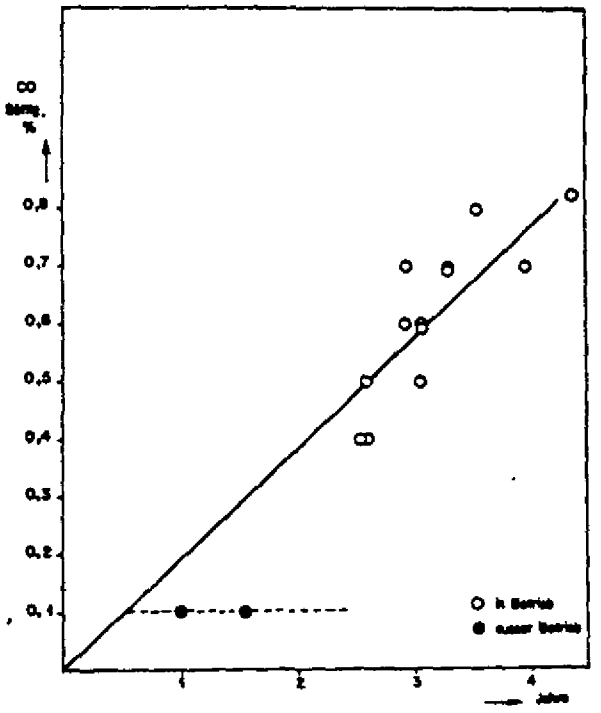

Figure 8 - Variation of the amount of CO_2 dissolved in the oil with the operating time or service life of the transformers
Sättig = saturation Jahre = year
o in operation
● at standstill

2.2. Gas formation in faulty operating transformers - Below the results of analysis are given of gas mixtures which occurred in the last eight years and in which it was possible to find the cause of the fault either by inspecting the transformer, or by special measures.

2.2.1. Examination of the gases in the collector relay (Buchholz protection) - The summary shows clearly that it is possible to differentiate between three types of oil decomposition on the basis of the analyses of gas separated out in the collector relay :

---

(1) In this figure the concentration of carbon monoxide is given in percentages of saturation. This magnitude makes possible data on the free gas with which the oil sample would be in equilibrium. There are data on total pressures and partial pressures of this gas, measured in atmospheres. Thus for example a total saturation of 100 % means that the oil sample would be at solution equilibrium with a gas of 1 atm , a saturation of 1 % for a definite component means that the partial pressure of this component in the equilibrium gas amounts to 0.01 atm.

1. Oil decomposition due to low-current partial discharges in gas occlusions under oil.

2. Oil decomposition due to discharges under oil.

3. Purely thermal decomposition of the oil.

For allocation to one of the three cases it is sufficient if we know the components of hydrogen, methane, acetylene and ethylene in the gas and then determine their ratios in the mixture. The allocation then takes place in accordance with the rules derived from the empirical material :

Re 1. a. : The gas which separates out contains hydrogen and methane.
The ratio of methane to hydrogen in the mixture is < 0.01 in parts by volume.
b. : Acetylene and ethylene cannot be detected.

Re 2. a. : The gas which separates out contains hydrogen, methane, acetylene and ethylene.
The ratio of acetylene to ethylene in the mixture is ≥ 1 in parts by volume.
b. : The ratio of methane to hydrogen in the mixture is ≤ 0.1 in parts by volume.

Re 3. a. : The gas which separates out contains hydrogen, methane, ethylene and often also acetylene.
The ratio of acetylene to ethylene in the mixture is < 1 in parts by volume.
b. : The ratio of methane to hydrogen in the mixture is > 0.1 in parts by volume.

Figure 9 shows the different results of analysis entered in a diagram, which gives the measured ratios of methane to hydrogen in the mixture and the corresponding values for acetylene and ethylene. For the three causes of failure named in the diagram three ranges can be fixed in which nearly all corresponding results of analysis are situated.

2.2.2. Examination of the gases dissolved in the oil - Since at least a part of the gases which are formed dissolve in the oil, dissolved gas can also be used for fault diagnosis. It has been found advisable to determine the dissolved gas concentrations in percentages of saturation, and [illegible] saturation values for methane and hydrogen, as well as for acetylene and ethylene. Then, in accordance with experience so far, the rules given above for the mixture ratios apply also for dissolved gases. This means that the composition of a gas that would be in solution equilibrium with the oil has similar characteristic properties for the cause of the fault, as the gases which collect in the relay.

In Figure 10 we have entered the values measured on oil samples in the case of faults, in the same manner as in Figure 9 for samples of gas separating out, [illegible] mixtures [illegible] straight lines for the individual [illegible]. The ranges which are circumscribed are the same as those in Figure 9, the crosses indicating the measured values.

2.2.3. The development of oxides of carbon - The carbon monoxide and carbon dioxide partial pressures in the gas from the collector relay give indications on the decomposition of solid insulation. In Figure 11 we give for the second case (discharges) and the third case (thermal decomposition) the measured partial pressures of both gases in atmospheres, combined in accordance with the position of the fault. In the case of discharges which affect the insulated conductors and main insulation, the carbon monoxide contents of the gas were high (2 to 35 % by volume of the gas) except for one fault during which the discharge from the weakly insulated branch passed to the neutral point. With discharges originating from the switch only low carbon monoxide contents were measured except for the case of damage in which an insulating sleeve was carbonised. In all gas formation which was due to discharges the amount of carbon dioxide in the gas was small.

With purely thermal decomposition phenomena much carbon dioxide was also produced in addition to high carbon monoxide concentrations, when the position of the fault was in the winding.

In the case of partial discharges in gas occlusions only the formation of hydrogen and methane was observed, and this was not accompanied by the development of oxides of carbon.

3. Discussion of the results and conclusions - When transformers were operating normally only the separating out of carbon monoxide was observed, which was assumed by Potthoff and

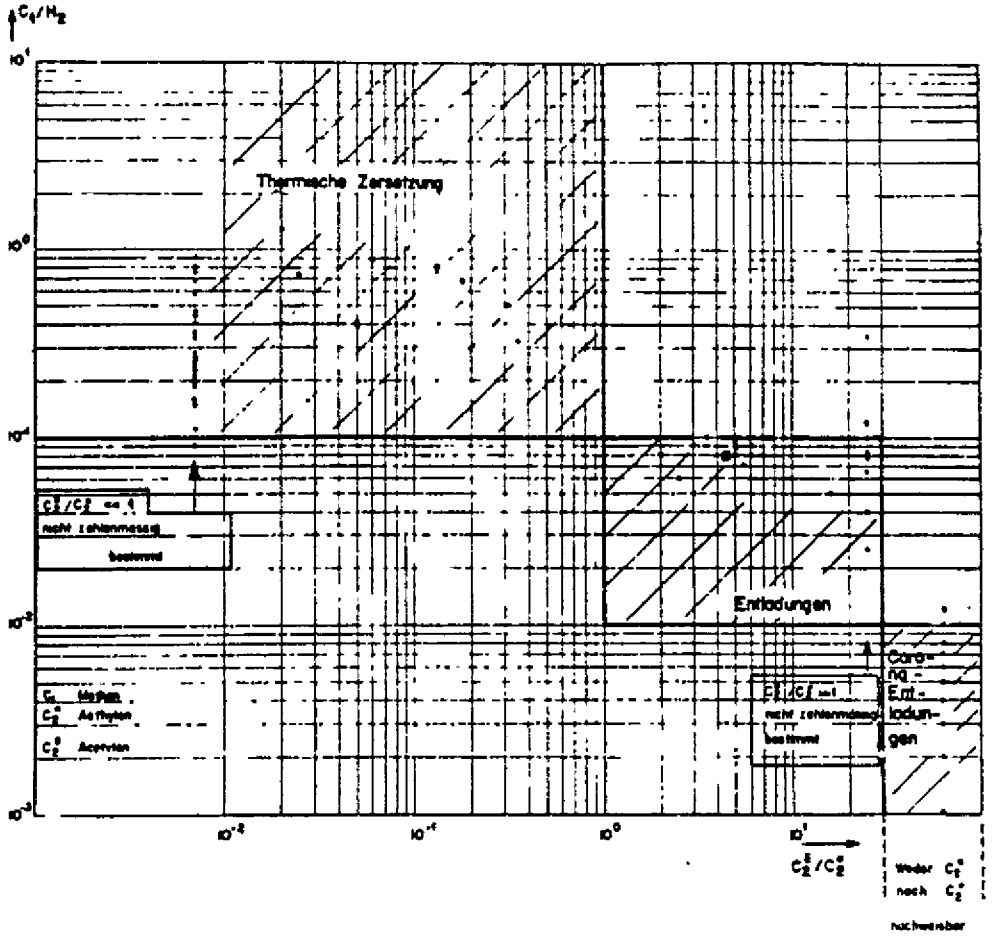

Figure 9 - Variation of the composition of the gas dissolved in the oil in the collector relays with the type of oil decomposition
Thermische Zersetzung = Thermal decomposition
nicht zahlenmässig bestimmt = not determined numerically
Entladungen . Discharges
Corona entladungen = Corona discharges
Weder $C_2^{=}$ = Neither $C_2^{=}$
Noch $C_2^{=}$ = nor $C_2^{=}$
Nachweisbar = detected

collaborators (Potthoff, K. and Uhrig, H., AEG-Mitt., 57, (1967), pp. 320 to 322). It was not yet possible to follow the simultaneous development of carbon dioxide.

For these three types of fault which have been listed the decomposition products of the insulating oil are typical. In spite of the differences due to the changing temperature distribution and the kinetics of the gas exchange between bubbles and oil in each separate case of damage, it is obvious that rules can be found in accordance with which the causes of faults can be divided up. In accordance with experience so far obtained these rules can also be applied to the decomposition products which are dissolved in the oil, if we work on the basis of the composition which the gas would have when in solution equilibrium with the oil.

In the case of the "creeping" gas generation it is obvious that the rules can be applied to the gases dissolved in the oil. Here all gas at first goes into solution, until the insulating liquid is saturated with gas (air plus decomposition gases) ; the exchange with the oil is therefore complete. Only when saturation is exceeded does gas separate out, and this is in equilibrium with the dissolved gas by reason of the complete exchange.

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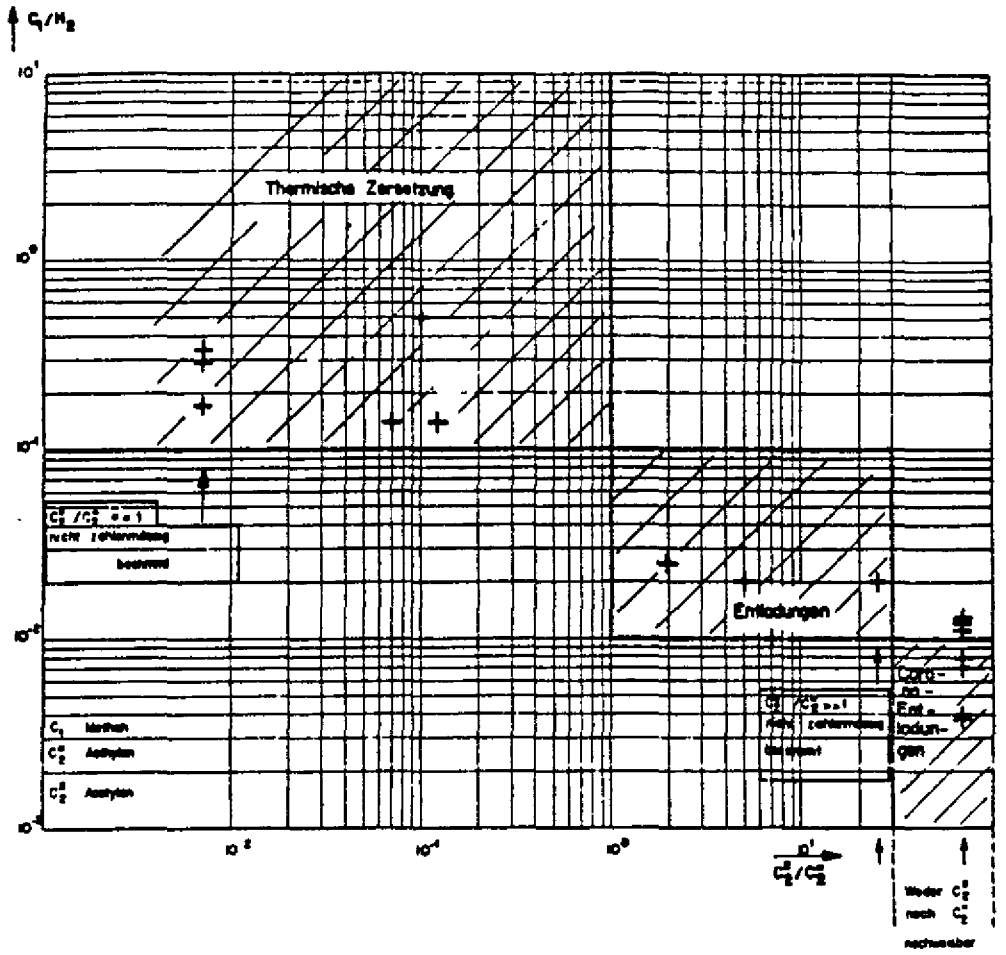

Figure 10 - Variation of the composition of the gases dissolved in the oil with the type of decomposition
Thermische Zersetzung = Thermal decomposition
Nicht zahlenmässig = not determined numerically
Entladungen = Discharges
Corona entladungen = Corona discharges
Weder C_2^{II} = neither C_2^{II}
Noch C_2^{III} = nor C_2^{III}
Nachweisbar = detected

But "creeping" processes are special cases and in the other cases equilibrium does not set in between gas and oil. Differences become apparent between dissolved and separated gas due to the kinetics of the gas exchange. The speed of exchange of the different gases depends on their solubility and diffusion coefficients in the oil. In the case of pairs of gases with approximately equal solubilities and diffusion coefficients, the exchange takes place at approximately the same speed. This means that the ratio in the mixture of a pair of gases of this type is not appreciably altered by the exchange. Acetylene and ethylene are a pair of gases of this type, and the rules based on the ratio of their mixture should be applicable equally for dissolved and free gas.

For the hydrogen and methane pair the exchange speeds are certainly different . if nevertheless the rules for free and dissolved gas state are approximately valid for their ratio of mixture, then this is due to the fact that it is mainly the solubility which determines the exchange speed, and that generally a relatively large oil volume is involved in the exchange with the gas bubbles.

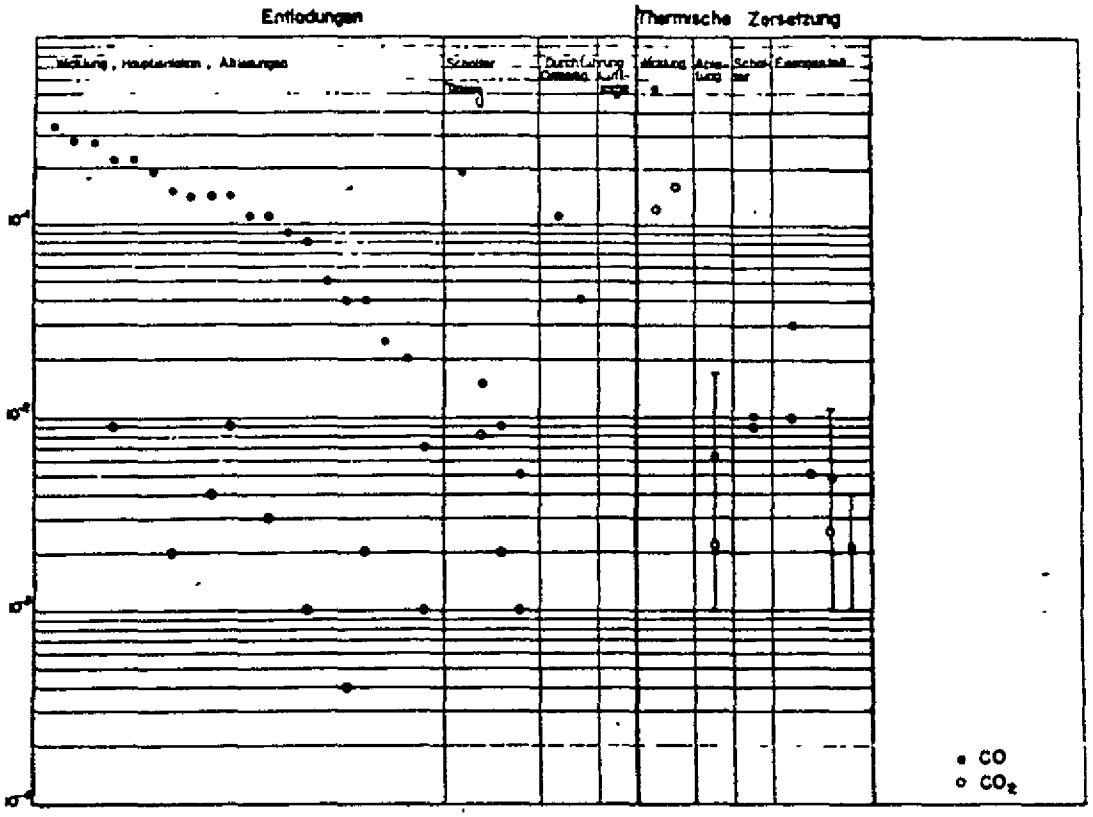

Figure 11 - Part by volume of carbon oxides (CO and CO_2) in the gases in collector relays of faulted transformers.
Entladungen = Discharges
Thermische Zersetzung = Thermal decomposition
Wicklung, Hauptisolation, Abteilungen = Winding, Main insulation, Conductor
Schalter = Isolator
Durchführung = Bushing
Oelseitig = Oil side
Luftseitig = Air side
Wicklung = Winding
Abteilung = Conductor
Eisengestell : Steel frame

Of course it is not possible to determine the fault point from the gas analysis In the case of purely thermal decomposition processes we can approach this aim if we follow the development of gas under different operating conditions. Gas separation during no-load operation, under different loads and different switch positions, then gives additional indications. On the other hand the possibility of locating discharges inside the transformer is small.

7. POSSIBLE EXTENSION TO THE CASE OF CABLES

The application of gaseous phase chromatography methods to the case of high-voltage cables with paper-oil insulation has been undertaken by the BICC Research Centre. Modifications were made to the classical methods with a view to increasing their sensitivity. In fact, due to the fact that the contents of humidity and oxygen in cables are much smaller than those encountered in transformers, the speeds of formation of the deterioration products are lower

MONS 204673

Askarel Guide

PROPOSED ASTM COMMITTEE D-27
DEFINITION AND SCOPE OF ASKARELS

DEFINITION

Askarel is a generic term for a group of non-flammable synthetic chlorinated aromatic hydrocarbons used as electrical insulating media.

~~SCOPE~~ General Characteristics of Liquids

Askarels of various compositional types are used. Under arcing conditions the gases produced, while consisting of predominantly non-combustible hydrogen chloride, can yield varying amounts of combustible gases, depending upon the askarel type.

Part II.

Insulation systems incorporating these askarels and cellulosic or other organic materials may, when arced, produce gaseous mixtures which are moderately flammable. As a precaution, such gases should be removed from the askarel by bubbling dry nitrogen through the askarel and flushing the gas space with dry nitrogen before any work is performed on the apparatus.

Note: ASTM is currently balloting the above proposed change. We should either agree to the above or propose changes. Obviously, ASTM and IEEE should use the same definition and scope and the one we have in the present askarel guide needs to be changed as per our discussion at the last meeting.

E. L. Raab

9-4-69

MONS 204674

Askarel Guide

Subject: Ballot on Proposed "Guide For Acceptance and Maintenance of Transformer Askarel In Equipment"

Pittsfield, Mass.
March 4, 1969

To: Members of I.E.E.E. Transformers Committee

Gentlemen:

Attached is a copy of the latest revision of the proposed "Guide For Acceptance and Maintenance of Transformer Askarel In Equipment" for your consideration and ballot. It is a modernized and enlarged version of AIEE No. 76 which was dated December 1958 and is the result of several years' effort by your Insulating Fluids Subcommittee.

It may be of interest to note that the present composition of the Insulating Fluids Subcommittee consists of six utility representatives, seven manufacturers' representatives and one general interest. At our November 1968 meeting in Philadelphia, the subject guide received the unanimous approval of all members of the subcommittee.

Please return the lower part of the enclosed ballot to the undersigned by April 14 at the latest as indicated on the ballot. Note that all ballots should be returned in order to satisfy the voting requirements demanded by Headquarters.

Very truly yours,

E. L. Raab lm L

E. L. Raab, Chairman
Insulating Fluids Subcommittee

ELR:ml

Att. (2)

Copies: Insulating Fluids Subcommittee Members

MONS 204675

I.E.E.E. Transformers Committee

Ballot on proposed "Guide For Acceptance and Maintenance of Transformer Askarel In Equipment"

I approve the proposed Guide *_____

I do not approve the proposed Guide **_____

NOT voting _____

*
Comments:_____

**
Please give reasons For Disapproval:_____

Retain Above For Your Record.

MONS 204676

GUIDE

FOR

ACCEPTANCE AND MAINTENANCE OF TRANSFORMER ASKAREL

IN EQUIPMENT

MONS 204677

ACKNOWLEDGEMENT

The Institute wishes to acknowledge its indebtedness to those who have so freely given their time and knowledge, and have conducted experimental work on which many of the I.E.E.E. publications are based.

The work of preparing the Guide was carried out by members of the Insulating Fluids Subcommittee of the I.E.E.E. Transformers Committee. The members of the subcommittee are:

E. L. Raab, Chairman

P. G. Benignus
G. L. Blenkle
J. E. Dind
D. A. Gillies
C. Keil
A. H. Locke
R. I. Lowe
W. H. Meade*
E. L. Morrison
V. R. Mulhall
J C. Parker
P. S. Pugh
C. Reinhardt
R. L. Schwab
T. K. Sloat
~~P. K. Sloat~~

*Deceased

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ASTM methods referred to in this Guide are obtainable at Headquarters of the American Society For Testing and Materials at 1916 Race Street, Philadelphia 3, Pa.

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GUIDE

OF THE

INSTITUTE OF ELECTRICAL AND ELECTRONICS ENGINEERS

ACCEPTANCE AND MAINTENANCE OF TRANSFORMER ASKAREL IN EQUIPMENT

I INTRODUCTION

The term askarel generally describes a widely used, broad class of non-flammable synthetic halogenated hydrocarbon insulating liquids. In this Guide, it applies solely to askarel in transformers, reactors, and accessory equipment operated at power frequencies.

Askarel contained in such apparatus as received from the manufacturer but prior to service operation should exhibit certain properties in order to insure satisfactory performance. It should be expected that askarel properly sampled from such equipment usually exhibits characteristics somewhat different from those obtained on new askarel which has not been in contact with apparatus constructional materials. Experience has indicated that variation of some characteristics of the askarel will not impair the service life of the equipment.

However, certain essential properties must be retained if askarel is to perform reliably its dual role of electrical insulation and heat-transfer agent.

It must have adequate dryness and a dielectric strength sufficient to withstand the electric stresses imposed in service. It must retain a sufficiently low viscosity so that its ability to circulate and transfer heat is not impaired. It should not be allowed to become so deteriorated or contaminated that it adversely affects the operation of the apparatus.

This guide assists the power equipment operator in evaluating askarel as received in transformers, reactors and accessory equipment operated at power frequencies and in his efforts to maintain askarel in serviceable condition. It recommends standardized tests and evaluation procedures. Methods are outlined for reconditioning and reclaiming askarel whenever necessary.

II ASKAREL - GENERAL CHARACTERISTICS

Commonly used transformer askarel consists of chlorinated aromatic hydrocarbons, and unlike mineral insulating oil is not subject to oxidation, is non-flammable, and is incapable of evolving explosive gases when decomposed by an electric arc. Consequently, data obtained from flash and fire point testing require somewhat different interpretation for the two fluids.

MONS 204680

In comparison to mineral insulating oil, askarel is a relatively polar ~~mineral~~; i.e., its molecules are dipoles, free to rotate around their axes and responsive to orientation by electrical forces. Askarel also exhibits a much higher dielectric constant ~~and capacitance~~ than insulating oil, and these differences must be kept in mind when interpreting electrical test data.

Because it is relatively polar, and possesses high solvency power, askarel is much more electrically sensitive to trace contaminants than insulating oil, and consequently the choice of constructional materials destined for use in askarel is very critical. This sensitivity is reflected in the power factor and resistivity (specific resistance) of the askarel.

It is important to note that, with the exception of water, the dielectric breakdown voltage of askarel is not generally adversely affected by many of the contaminants to which its power factor and resistivity (specific resistance) are so sensitive. In fact, the dielectric breakdown voltage of askarel is somewhat greater than that of insulating oil. Therefore, the values assigned these dielectric fluids in newly supplied transformers are 30 kV min. and 26 kV min., respectively.

As with insulating oil, askarel must be kept dry. It can pick up moisture from exposure to humid atmosphere. Under similar conditions of exposure, askarel can pick up nearly twice as much moisture when measured on a parts per million weight basis.

A final obvious difference is that askarel is about one and one half times as heavy as water, whereas mineral oil is lighter than water. This means that any free water present will float on askarel whereas free water in oil will be at the bottom.

III EVALUATION OF ASKAREL RECEIVED IN NEW EQUIPMENT

Some users of askarel equipment find it desirable to make "as received tests" on all equipment. It is quite common to use the dielectric breakdown voltage test and visual appearance as the most significant tests. If suitable equipment and trained personnel are available additional information may be obtained from the power factor, color, and moisture content tests.

In sampling askarel contained in apparatus extreme care must be exercised in order to obtain a representative sample. ASTM Method D 923 should be followed.

New equipment with askarel exhibiting the following characteristics is considered acceptable:

		ASTM Methods
Dielectric Breakdown Voltage	30 kV min.	D 877
Color	300 max. (Straw color)	D 2129
Condition - Visual	Clear	D 1702
Water Content	35 parts per million max.	D 1533
Power Factor at 25C	*	D 924, D 150

PF

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*The power factor of askarel taken from new transformers, reactors and accessory equipment can reflect the presence of moisture, dissolved polar compounds, or other contaminants and may vary with the type of equipment from which the sample was taken due to the different ratios of liquid-to-solid insulation and to the high solvency power of the askarel. Given an acceptable water content and dielectric breakdown voltage of the askarel as indicated above, a high power factor seldom impairs the serviceability of the askarel within rather broad limits and is indicative of the degree of contamination present.

Recognizing the possibility of a wide range of power factors being recorded for askarel in new equipment, it is difficult to establish a single limit which would be acceptable to both suppliers and users for all applications. However, as a broad guide, power factors up to about 10% at 25C and 60 cycles per second (hertz) do not in general indicate any abnormal contamination providing that the other criteria (water content, dielectric breakdown voltage, etc.) are met. Much higher power factors may indicate excessive contamination or the misapplication of the solid materials used in manufacture in contact with the askarel and should be investigated.

The significance of the foregoing and other tests listed in this guide are discussed in Section VII.

IV CLASSIFICATION OF SERVICE AGED ASKAREL

It is extremely difficult, if not impossible, to indicate the value of specific tests and recommended test limits for all possible existing applications of askarel. It should also be recognized that with the present state of knowledge no one test can be used as the sole criterion of condition of askarel. It is possible, however, to summarize the value and importance of current tests and to suggest methods of treatment for the askarel being examined, such methods being based on current industry experience. Askarel may be placed in the following classifications based upon composite evaluation of significant characteristics and on field and laboratory screening tests.

Group I

This group contains askarel which is in satisfactory condition for continued use.

Group II

This group contains askarel which requires only minor reconditioning for further service. (Reconditioning is the removal of moisture and insoluble contaminants. The usual method employed is filtration.)

Group III

This group contains askarel in poor condition. It should be reclaimed or discarded depending upon economic considerations. (Reclamation involves the use of methods and processes which result in a purification of the askarel. Treatment with an adsorbing agent such as fuller's earth is most widely used.)

Group IV

This group contains askarel in such poor condition and requiring such drastic treatment that it is not feasible either technically or economically to attempt reclamation. Examples of such a condition would be a severely arced or excessively oil contaminated askarel.

V ECONOMIC FACTORS

In this section of the Guide, askarel is classified on the basis of significant characteristics. Many of the undesirable characteristics of used askarel can be corrected by reconditioning or reclaiming, if economic conditions justify it. The alternate choice is to replace with new askarel.

A review of the experiences of many users of electric equipment reveals a wide variation in the actual cost data involved in the reconditioning or reclaiming of askarel. This variation can be attributed to the amount and condition of askarel involved, laboratory and shop facilities, whether the work is performed at one central or several distant locations and the availability of qualified personnel.

Askarel that contains water and insoluble contaminants can be reconditioned by mechanical means using various filter devices. If soluble and/or colloidal contaminants are present in the askarel, reclaiming can be done with special filtering devices or a special filter press using a material such as fuller's earth.

To determine if reconditioning or reclaiming of askarel is economically justifiable, a number of factors must be considered. Some of these factors are as follows:

1. Value of askarel being considered for processing
2. Cost of processing materials
3. Total cost of process vs. quality of end-product
4. Equipment maintenance and amortization
5. Cost of collection and storage
6. Labor and transportation costs
7. Laboratory cost
8. Loss of askarel during processing
9. Cost of scavengers and make-up of chemicals if addition is necessary and desirable
10. Cost of disposing of processing material and scrap askarel
11. Cost of new askarel delivered at job site

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VI SAMPLING

Representative samples, whether of the complete contents or only parts thereof, are extremely important from the standpoint of evaluation of the quality of the product sampled. Obviously careless sampling procedure or contamination in the sampling equipment will result in a sample that is not truly representative. This generally leads to erroneous conclusions concerning quality and incurs loss of time, effort, and expense involved in securing, transporting, and testing the sample. It is strongly recommended that all of the procedures and precautions outlined in the latest revision of ASTM D 923 (Sampling Electrical Insulating Liquids) be followed.

Because of the high specific gravity (relative density) of askarel (greater than 1), water and some other impurities are most likely to be found at or near the surface. The top sample, therefore, is considered to represent the worst condition.

VII ASTM ASKAREL TESTS AND THEIR SIGNIFICANCE

There are a number of tests that can be applied to askarels as a basis for their classification as indicated in the foregoing. It is recommended, wherever possible, that tests be the latest revisions accepted as standards or tentatives by ASTM. These tests and their significance are as follows:-

		ASTM Methods
1.	Color	D 2129
2.	Dielectric Breakdown Voltage	D 877
3.	Flash and Fire Point	D 92
4.	Inorganic Chlorides	D 1821
5.	Neutralization Number	D 974, D 664
6.	Pour Point	D 97
7.	Power Factor	D 924, D 150
8.	Refractive Index	D 1807
9.	Resistivity (Specific Resistance)	D 1169
10.	Scavenger Content	D 1701
11.	Specific Gravity (Relative Density)	D 1810
12.	Viscosity	D 88, D 445
13.	Visual Examination of Service Aged Askarels in the Field	D 1702
14.	Volume of Oil in Oil-Contaminated Askarel	D 1808
15.	Water Content	D 1533

1. Color - D 2129

The color of askarel is a numerical value based on comparison with a series of numbered color standards with transmitted light under prescribed conditions. Color is chiefly significant as an indicator of contamination in askarel. Frequently this contamination is the result of solvent action between the askarel and other materials in the apparatus involved. No definite relationship can be established between color and the physical and electrical characteristics of the liquid.

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2. Dielectric Breakdown Voltage - D 877

The dielectric breakdown voltage of askarel may be defined as the voltage at which electrical breakdown of the material occurs under prescribed conditions. The dielectric breakdown voltage of askarel is of importance as a measure of its ability to withstand electrical stress without failure. It may also indicate the presence of contaminating materials, such as water, conducting particles, dissolved contaminants, or the decomposition products resulting from an electric arc. A high dielectric breakdown voltage, however, is not a certain indication of the absence of all contaminants.

3. Flash and Fire Point - D 92

The flash point of askarel is the temperature to which askarel must be heated in order to give off sufficient vapor to form a flammable mixture with air under the conditions of the test. Askarels have a flash point which is not indicative of a hazardous condition unless followed by a fire point.

The fire point of a material that emits combustible vapor may be defined as the temperature to which the material must be heated in order to support continuous combustion when exposed to the atmosphere under prescribed conditions. Askarels have no fire point. The test is only of value, therefore, in showing whether the material has the nonflammable characteristics required of an askarel, or has a large amount of combustible contaminant.

4. Inorganic Chlorides - D 1821

In the presence of water chlorides can ionize. Their corrosive action can then be detrimental to the life of the apparatus in which the askarel is used. The presence of ionizable chlorides may be indicative of arc decomposition of the askarel.

5. Neutralization Number - D 974, D 664

In the inspection of new askarels, the neutralization value is of importance as a quality index of purity.

Since askarel is not subject to deterioration by oxidation, small changes in the neutralization value of service aged askarels may indicate the solution of basic or acidic materials from the various solid materials in contact with the askarel or the deterioration of such soluble materials to form basic or acidic materials. A large change in acidity may indicate decomposition of the askarel by an electric arc.

6. Pour Point - D 97

The pour point of askarel may be defined as the temperature at which the liquid just flows under prescribed conditions of test. The pour point has little significance as far as contamination or deterioration is concerned but may be used for type identification.

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7. Power Factor - D 924, D 150

Power factor is the ratio of the power dissipated in the askarel in watts to the product of the effective voltage and current in volt-amperes, when tested with a sinusoidal field under prescribed conditions. Since askarel is not subject to oxidation, an increase of power factor value of the askarel in service may be attributed to the presence of moisture, dissolved polar compounds or other contaminants. Depending upon the type of apparatus and application, high power factor due to contamination other than moisture seldom impairs the serviceability of the askarel. However, the high power factor may influence the transformer winding power factor.

8. Refractive Index - D 1807

Refractive Index is defined as the ratio of the velocity of light in air to its velocity in the substance under prescribed conditions. The refractive index of askarel varies with its composition and with the nature and amount of contaminants held in solution. Changes of refractive index of askarel in service may be useful in estimating compositional change and degree of soluble contamination.

9. Resistivity (Specific Resistance) - D 1169

Volume resistivity (specific resistance), in ohm-centimeters, of askarel is the ratio of the dc potential gradient in volts per centimeter paralleling the current flow within the sample to the current density in amperes per square centimeter at a given instant of time and under prescribed conditions. The resistivity of askarel is a measure of its electrical insulating properties. High resistivity normally reflects low content of free ions and ion-forming particles and indicates a low concentration of conductive contaminants.

10. Scavenger Content - D 1701

Askarels are decomposed by an electric arc with the evolution of HCl gas. A scavenger, which reacts chemically with dissolved HCl gas to form a nonvolatile reaction product, may be added to an askarel and serves a useful function in improved preservation of the submerged portion of the apparatus should an arc occur. Measurement of scavenger content indicates the amount of protection available against dissolved arc-formed gases and permits estimation of the amount of make-up additive required.

11. Specific Gravity (Relative Density) - D 1810

The specific gravity (relative density) of an askarel is the ratio of the weights of equal volumes of askarel and water at 15.56C (60F). Specific gravity (relative density) may be useful for type identification or to determine marked compositional changes.

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12. Viscosity - D 88, D 445

The viscosity of an askarel is its resistance to uniformly continuous flow, without turbulence, inertia, or other forces, and is usually determined by measuring the time of flow of a given quality of liquid under controlled conditions. Viscosity of askarels varies with temperature, and for that reason is always designated at a specified temperature.

Viscosity is a controlling factor in the dissipation of heat by convection in insulating and cooling liquids. This is particularly important in transformers and other apparatus where heat generated in windings and cores must be largely removed by transmission through the liquid to a heat exchanger or the containing case. It is also a factor in the rate of impregnation of certain apparatus.

13. Visual Examination of Service Aged Askarels In The Field - D 1702

This method provides for field examination of service aged askarels, sampled from equipment in service, chiefly for the purpose of determining whether laboratory tests are required. This method may be of significance in visually detecting cloudiness which is an indication of the presence of moisture, carbon particles, which may show an arcing condition, or color which is chiefly significant as an indicator of contamination in askarels. No definite relationship has been established between color and the physical and electrical characteristics of the liquid. By the use of this method it may be possible to extend the period between routine laboratory tests.

14. Volume of Oil in Oil-Contaminated Askarel - D 1808

The quantitative determination of the volume of oil in oil-contaminated askarel is important as a check on the degree of contamination and as a means of ascertaining when the nonflammable property of this fluid has been impaired to such an extent that it can no longer be classified as a nonflammable liquid.

15. Water Content - D 1533

The water content is that amount of water, expressed in parts per million by weight, which is present in the liquid. The test is significant in that it will show the presence of water which may not be evident from electrical tests. Changes in water content of askarel in service may be indicative of undesirable operating conditions requiring correction.

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VIII TESTING PROCEDURES

The testing of askarels is best accomplished in adequately equipped laboratories. However, some find it advantageous to make "Field Screening Tests".

(A) Field Screening

Field screening tests are usually made to determine which samples of askarel may require laboratory investigation. Since most tests of askarel can be done best with laboratory equipment and trained personnel, field tests are usually limited to visual inspection and dielectric breakdown voltage.

No standardized procedure is available at the present time for performing field tests on service aged askarel or for classification. Portable dielectric test sets have been available for some time and have proved satisfactory. In addition to the above tests, some operators with suitable field equipment and trained personnel may wish to gain additional information by use of a power factor test. In interpreting results of these tests, consideration must be given to other characteristics of the askarel.

(B) Laboratory Screening

Askarel requiring laboratory evaluation will vary widely in condition depending upon the degree of contamination. There are many tests of a research nature which could be applied, but the general use of such tests would be impractical. The following tests, therefore, are believed to be adequate for classifying service aged askarel.

1. Dielectric breakdown voltage
2. Inorganic chlorides
3. Neutralization number
4. Power factor or resistivity (specific resistance)
5. Specific gravity (relative density)
6. Visual condition and color
7. Water content

IX RECONDITIONING AND RECLAIMING SERVICE AGED ASKAREL

A. General

Askarel does not oxidize, sludge, or decompose in normal transformer use. It will break down when subjected to an electrical arc. If the arc exposure has been of long duration, it is usually best to discard the fluid.

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However, when arc exposure has been relatively mild or if the askarel is merely contaminated with moisture and other conducting impurities, restoration is practical. An exception is the lack of a practical method for removing mineral oil from askarel. Askarel contaminated with over 2% by volume of transformer mineral oil may no longer be classified as a nonflammable liquid.

B. Treatment After Exposure to An Arc

When askarel is exposed to arcing, hydrogen chloride gas is liberated. Some hydrogen chloride will remain dissolved in the askarel, and must be removed as quickly as possible.

This is done by bubbling about 220 cubic feet (6.23 cubic meters) (1 cylinder) of dry nitrogen through each 100 gallons (3.785 cubic meters) of askarel. The nitrogen should be admitted at the bottom of the tank or container vented sufficiently to allow the nitrogen to escape, while preventing entrance of moisture. This procedure for removing hydrogen chloride is recommended even though the askarel contains a hydrogen chloride scavenger.

C. Filtering Through Dry Blotter Paper to Remove Moisture and Extraneous Particles

Since undissolved moisture will be at the surface of the askarel any simple means should be used to drain or siphon off excess water.

The final drying is done by circulating the askarel through dry blotter paper. The askarel temperature should not exceed 60C. Several passes through multiple layers of freshly dried paper may be required. Filtering should not be done when the relative humidity exceeds 75%.

The filter paper must be dried immediately before use. For best results, the paper is spread for maximum surface exposure in a hot air circulating oven and heated for 4 - 6 hours at 110C.

After filtration the dielectric breakdown voltage of the askarel should be at least 35 kV.

D. Fuller's Earth Treatment for Maximum Improvement of Power Factor and Volume-Resistivity

The askarel should be relatively dry prior to final refinement with conditioned fuller's earth.

Most operators prefer a portable fuller's earth refining apparatus such as a plate press or an earthen cartridge type filter. As this apparatus involves recirculation, the askarel should be removed from the top of the tank and returned at the bottom, because free moisture and most of

the other undissolved contaminants will be found near the surface of the askarel. Since these recirculating procedures can introduce air into the askarel, sufficient time (at least 4 hours and preferably over night) should be allowed for the air to escape before re-energizing the apparatus.

When a filter press is used, the plates are fitted with dry filter paper. This is coated with 0.1 to 0.2% by weight of dried fuller's earth based on the weight of askarel to be treated. (Askarel weighs nearly 13 pounds per U. S. gallon.) (1556 Kilograms per cubic meter) The finely divided clay should be activated and dried to not over 1% of moisture by heating for 12 hours in shallow trays at about 200C, immediately prior to use.

To deposit the fuller's earth evenly, about 1/3 of the total quantity is stirred into a small portion of the askarel in a clean container. This mixture is pumped through the filter, then followed with two more 1/3 portions. Then the askarel is pumped from the top level of the tank, through the filter and returned to the tank at the bottom. It is desirable that the askarel temperature not exceed 60C during the filtering operation.

Circulation is continued until the fluid is clear and tests show that the electrical properties are fully restored.

E. Addition of Scavengers

Only slight loss (by selective absorption) of the hydrogen chloride scavengers occurs when askarel is refined by treatment with 0.2% by weight of fuller'e earth at a temperature not above 60C. Significant removal of the additives can result from treatment with much larger amounts of fuller's earth.

Initially 0.1 - 0.2% by weight of scavenger, usually an epoxide, is incorporated in modern transformer askarel. Inquiries about replenishing the additive should be directed to the transformer manufacturer, or to the supplier of the fluid.

X HANDLING MATERIALS

All common metals are satisfactory for use with askarel in dry environments. To avoid rust, it is preferable to use stainless steel or aluminum for filter presses or equipment used intermittently or in the presence of moisture.

Because stainless steel piping may be difficult to fabricate, the use of aluminum or galvanized steel is more common.

Pipe connections should be welded wherever possible. Flanged connections are very good when fitted with a sufficient number of bolts to hold the gasket seal securely.

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Where readily removable connections are needed, deep pipe threads should be cut and wrapped with Teflon tape to make the seal.

In addition to Teflon, other materials completely compatible with askarel and suitable for gaskets are: Viton and Silastic (silicone) elastomers and Flexitallic stainless steel rings with either Viton, Teflon or silicone elastomer between the steel laminations. Silicone elastomer does not have good abrasive resistance and it is not suitable for use with mineral oil Viton has relatively good abrasion resistance and is resistant to both askarel and mineral oil.

If a fine grain cork-nitrile rubber composition gasket is used, it is desirable to coat the exterior of the seal with room temperature curing epoxy cement or RTV silicone cement.

Well cured or dried adhesive cements of dewaxed orange shellac, dextrine glue, Elvanol (polyvinyl alcohol acetate), phenolic or epoxy, are relatively noncontaminating to askarel.

Suitable hoses are those of the flexible metal type, or those in which only Teflon or silicone polymers are in contact with the askarel.

The preferred valves are those in which only stainless steel is in contact with the askarel. Where required, the stainless steel valve may be seated with Teflon. Also brass valves are acceptable.

Suitable valve stem packing is the #117 braided Garlock type, or Raybestos Manhattan type 365-C, or any equivalent asbestos graphite packing.

Centrifugal pumps suitable for handling hot fluids are the recommended type. Gear type pumps are not desirable. All wetted surfaces of the pump should be of stainless steel. The shaft seal should be the external mechanical (carbon ring) type to eliminate packing material and its exposure to askarel. All askarel handling equipment should be reserved solely for use with askarel, and should not be used interchangeably with mineral oil.

XI ASKAREL USED UNDER MILD ARCING CONDITIONS

Askarel is used, to some extent, in apparatus where it is subjected to light intermittent arcing, such as in self-contained induction regulators, where operating switches are continually producing slight arcs, in transformer deenergizing switches, etc. Under normal operating conditions, deterioration of the askarel is very slight. However, improperly adjusted or defective switches in this type of apparatus can produce excessive and prolonged arcing and accelerated deterioration of the askarel. It is recommended that when askarel is used under these conditions, checks of the liquid, especially for moisture and dielectric strength, be made more frequently than when it is used only as a cooling and insulating fluid. Deterioration of this type is indicated by a blackening of the liquid. It can usually be reconditioned as previously described. Special attention should be given to maintaining the scavenger at the appropriate concentration.

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XII ASKAREL UNDER EXCESSIVE TEMPERATURE OR FAULT CONDITIONS

Chlorobenzenes used in transformer askarels begin to boil at temperatures of about 205C, under atmospheric conditions. If the material is heated to such high temperature in a sealed system, pressure develops. Pressure will also develop in the system if the askarel is arced sufficiently to generate copious hydrogen chloride gas.

Therefore, it is recommended that wherever possible, sealed askarel filled equipment be provided with pressure relief devices. These devices must be large enough to provide immediate relief at a definite pressure, and to prevent further build up of pressure if decomposition continues. It must be remembered that the presence of devices of this sort does not necessarily preclude the rupturing of containing vessels, since pressure build up can be extremely rapid under violent arcing conditions.

XIII RECOMMENDED HANDLING PRECAUTIONS

It is generally accepted that the exposure to askarel is not hazardous, provided simple precautions are taken. Most people can handle askarel in the same manner as insulating oil without fear of adverse effects. Some people are allergic to askarel, and continued exposure may result in skin irritation. Therefore, direct contact with askarel and its vapors, particularly when hot, should be avoided. This combined with personal cleanliness should constitute adequate safeguards against harmful effects. Eye protection (glasses, shields, etc.) are required. The use of any type glove should be avoided. Medicinal washes or detergents followed by an application of cold cream or Silicare have been successfully used to eliminate irritation resulting from askarel coming into contact with an open cut or skin abrasion. A drop of castor oil has been found to neutralize, in most cases, irritation caused by contact of askarel with the eyes.

Exposure to concentrated askarel vapors should be avoided, particularly in closed, unventilated rooms. As with many volatile liquids, the vaporization of askarel increases rapidly with temperature. Therefore, ventilation which is adequate for handling askarel at room temperature may not be adequate for handling askarel at elevated temperatures. The gases produced when askarel is decomposed by very high temperatures or an electric arc in the presence of air or organic insulating materials, contain a high percentage of hydrogen chloride, and small percentages of carbon dioxide, carbon monoxide and oxygen. Very small concentrations of this combination of gases are very unpleasant and irritating, thus giving ample warning of their presence. The all-purpose canister type gas masks are suitable for protection from gases liberated from decomposed askarel.

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XIV STORAGE AND HANDLING

Transformer askarel can be handled in about the same manner as transformer mineral oil.

A. Drums

The askarel drum is made of steel lined with a specially selected material properly cured. Galvanized drums may also be used.

Askarel must be kept dry. It can pick up moisture from contact with humid air. Drums of askarel should not be stored outdoors in an upright position, as water can collect in the dish of the drum head, and can be drawn into the drum under certain conditions. Drums should be stored on their sides with the bungs downward and under cover. It is preferable to store drums indoors.

Askarel should not be transferred into drums that have previously contained other materials.

B. Tank Cars

Tank cars are solid aluminum, or steel lined with aluminum, or a zinc-tin coating.

It is desirable to provide canopies for unloading askarel cars. The car should not be opened in the rain or snow. It is desirable to not open the car if the relative humidity exceeds 75%. Precautions must always be taken to exclude dirt.

C. Sampling

(Please refer to Section VI)

D. Effect of Light Exposure

Prolonged exposure to ultraviolet light (sunlight, some fluorescent lights) should be avoided. Askarel can decompose under such conditions. Sample bottles are preferably of the amber glass type.

XV UNDERWRITERS' LABORATORIES REPORTS

The Underwriters' Laboratories have exhaustively examined, at the electrical manufacturers' requests, the miscellaneous hazards involved in the use of askarel and askarel-filled apparatus. Reports have been issued to the respective manufacturers covering the tradenamed askarels being considered. It is suggested that interested users of transformer askarel contact the manufacturers for this information.

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APPENDIX

RESULTS OF SURVEY OF UNITED STATES AND CANADIAN USERS OF TRANSFORMER ASKAREL

I GENERAL

This survey of the United States and Canadian users of askarels was made in 1966 in order to show the practices followed in testing, reconditioning, and reclaiming of askarels. Of the 96 inquiries sent out, 50 replies were received with usable information which is a 52% return. All of the following data are shown as a percentage of those returning the questionnaires and it should be pointed out that this includes information from both manufacturers as well as users and no attempt has been made to classify them according to the size of the systems involved.

Thirty-two percent of the users make only field tests; thirty-two percent make only laboratory tests; twenty-four percent make both field and laboratory tests; and twelve percent make no tests at all on askarels. Most all make tests at a fixed time interval and the largest group follow a yearly schedule. Sixty-seven percent recondition askarel which is below acceptable limits, and the most popular method employs the combination fuller's earth and blotter press.

This survey was made purposely to determine the types of tests being made by askarel users and to give an indication of acceptable limits. The following data are the over-all results of the questionnaire.

Askarel - Field Tests

Fifty-six percent of askarel users do some type of field testing. Of these 93% make a test for dielectric breakdown voltage and 57% make at least two field tests;

(a) Dielectric breakdown voltage
(b) Visual examination and color

Other tests used in decreasing order of frequency are power factor, neutralization number and moisture.

Askarel - Laboratory Testing

Fifty-six percent of askarel users make an average of four tests in their laboratories on samples. Of these 93% determine the dielectric breakdown voltage and

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of the following make at least four tests:

(a) Dielectric breakdown voltage
(b) Color and condition
(c) Neutralization Number
(d) Moisture content
(e) Power factor

Other tests used in decreasing order of frequency are inorganic chlorides, resistivity (specific resistance) and specific gravity (relative density).

Askarel - New Equipment

Eighty percent of the users test the dielectric breakdown voltage of askarel in new equipment. Twenty-two percent also test:

(a) Power factor
(b) Moisture

Other tests used in decreasing order of frequency are neutralization number, inorganic chlorides and resistivity (specific resistance).

II TESTING

A. [illegible] reporting tests, [illegible];

1. 32% use only field tests
2. 32% use only laboratory tests
3. 24% use both field and laboratory tests
4. 12% do not test

B. The relative importance of the field tests are as follows:

1. 93% dielectric breakdown voltage
2. 40% color and visual examination
3. 14% power factor
4. 11% neutralization number

C. The average limits on service aged samples are as follows:

1. Dielectric breakdown voltage - Average 27 kV - Range 20-35 kV
2. Power factor - Average 10% - Range 2-40%
3. Neutralization number - Average .12 - Range .05-.5
4. Inorganic chlorides - Average .5 - Range .01-2.0
5. Moisture - Average 45 ppm - Range 40-55 ppm

D. The average limits on askarel in new equipment are as follows:

1. Dielectric breakdown voltage - Average 30 kV - Range 22.5-35 kV
2. Power factor - Average 3% - Range .05-5%
3. Moisture - Average 32.5 ppm - Range 20-50 ppm

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III RECONDITIONING

A. 67% recondition askarel from transformers:

1. 73.5% prefer the combination fuller's earth-blotter press
2. 17.5% use only the blotter press
3. 9% use only fuller's earth

B. The average limits on reconditioned askarels are as follows:

1. Dielectric breakdown voltage - Average 29 kV - Range 23-35 kV
2. Power factor - Average 3.5% - Range 2-15%
3. Neutralization Number - Average .10 - Range .01-.5
4. Moisture - Average 35 ppm - Range 30-50 ppm

IV FREQUENCY OF TESTING

A. The frequency of testing practiced by askarel users is as follows:

1. 53% test every year
2. 17.5% test every five years
3. 17.5% test when there is a reason for testing
4. 9% test every two years
5. 3% test every six months

B. Most users test all askarel units at the same interval regardless of rating.

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March 21, 1972

STANDARD METHOD FOR DETERMINATION OF COMBUSTIBLE GASES IN ELECTRICAL APPARATUS IN THE FIELD

INTRODUCTION

Arcing, corona discharge and localized overheating in the insulation system of transformers will result in chemical decomposition of the insulating oil and other insulating materials with the formation of various gases. Portable combustible gas meters are a convenient means of detecting and estimating the amount of combustible gases and frequently give the first indication of a malfunction.

1. Scope

1.1 This method covers the detection and estimation of combustible gases in the gas blanket above the oil or in gas detector relays in transformers using portable field instruments. It is only applicable with transformers using mineral oil as the dielectric fluid. Gases dissolved in the oil and noncombustible gases are not determined. A method of calibrating the instruments with a known gas mixture is included.

1.2 This method affords a rough quantitative estimate of the total combustible gases present in a gas mixture. If a more accurate determination of the total amount of combustible gases or a quantitative determination of the individual components is desired, a laboratory analytical method using a gas chromatograph or mass spectrometer should be used.

2. Summary of Method

2.1 A sample of gas is diluted to a fixed ratio with air and introduced into the meter at a pressure of approximately one atmosphere. Any combustible gases present are catalytically oxidized on the surface of an incandescent platinum alloy wire

which is an element of a wheatstone bridge. When combustible gases oxidize on the surface they increase the temperature of the element which changes its resistance and upsets the balance of the bridge.

The change in the resistance of the indicating elements in the bridge circuit is indicated on a meter which is usually calibrated to read in percent total combustible gas.

3. Significance

3.1 Normal operation of a transformer may result in the formation of some combustible gases. The detection of an incipient fault, if present, involves an evaluation of the amount of combustible gas present, the rate of generation of these gases and their rate of escape from the transformer.

4. Apparatus

4.1 Portable Combustible Gas Meter[1] consisting of the following components:

4.1.1 Platinum alloy catalytic oxidizing elements incorporated in a wheatstone bridge circuit complete with a suitable battery and controls for balancing and voltage adjustment.

4.1.2 A milliampere meter preferably calibrated to read directly in percent total combustible gases.

[1]The following commercial instruments have been found to be satisfactory:

Davis Vapotester Model MS-1, Scott Aviation - Davis Products, 225 Erie Street, Lancaster, New York 14086.

J. W. Portable Indicator Model SS-P, Johnson-Williams Division of Bacharach Industrial Instrument Company, 2300 Leghorn Street, Mountain View, Californ

Indicator, combustible-gas, Explosimeter Model No. 2, Mine Safety Appliance Company, Pittsburgh, Pennsylvania.

Portable Fault-Gas Detector Model A, Pennsylvania Transformer Division, McGraw Edison Company, Canonsburg, Pennsylvania.

4.1.3 Gas mixing device for mixing air and sample gas at fixed ratios.

4.2 Standard Reference Gas

4.2.1 The Standard Reference Gas shall be a mixture of methane and nitrogen, with the methane content usually between 2 and 3 percent. The methane content should be known to the nearest 0.1 percent.

5. Safety Precautions

5.1 Gases generated in transformers can be explosive. The combustible gas meter should, therefore, be equipped with a flame arrester. Air should be used to dilute the sample gas. To avoid the possibility of an explosion, never use pure oxygen for this purpose. Normal precautions should be observed when sampling the gases from the transformer.

6. General Considerations

6.1 In this method it is essential that sufficient oxygen be present in the gas mixture to oxidize the combustible gases. Since the gas blanket in a transformer is usually an inert gas, it is necessary to dilute the sample gas with a known amount of air. This is usually accomplished by either introducing air and the sample gas into the instrument in known ratios through fixed orifices, or by mixing known quantities of air and sample gas externally by displacement over water before introduction into the instrument. The working range of these instruments is between the low limit of sensitivity and about the lower explosive limit. They generally read off scale at the high end between the lower explosive limit and the upper explosive limit and may indicate zero when the combustible gas content is above the upper explosive limit.

6.2 Contamination on the surface of the platinum alloy elements can seriously impair the sensitivity and response of the meter. This loss of response does not affect the normal balancing of the bridge circuit and is not detected by an electrical check of the instrument. It is, therefore, essential to check and calibrate the instrument frequently against a standard combustible gas mixture.

6.3 Since each combustible gas has its own individual instrument response, the values determined by an instrument are dependent upon the kind of combustible gas used for calibration. In order to permit interlaboratory comparisons of results, a mixture of methane in nitrogen has been selected as the standard calibrating gas mixture.

7. Calibration

7.1 Prepare the instrument for operation and make zero balance and voltage adjustments in accordance with the instructions of the instrument manufacturer and the Procedure, Section 8.

7.2 Mix the standard reference gas with air and introduce it into the meter in exactly the same manner as used for the sample gas from the transformer.

7.3 Turn the calibration adjustment so the meter indicates the known methane content of the reference standard when the air and reference gas mixture is being tested. Purge the meter with air to remove traces of reference gas and proceed as in Section 8.

8. Procedure

8.1 Prepare the instrument for operation in accordance with the instrument manufacturer's instructions. This should normally include a warm-up period to allow the battery voltage to stabilize, a voltage adjustment and an instrument balance or zero adjustment.

8.2 Calibrate the instrument against the standard methane mixture following the procedure in 7. The frequency of calibration required for individual instruments can be determined by experience. Low readings because of contamination on the platinum elements can only be detected by a check with a gas of known composition. In any case of doubt the instrument should be recalibrated.

8.3 If the instrument has a range switch or more than one air dilution orifice, set for maximum range or dilution to avoid damage to the instrument. Purge the instrument so it is free of gas from previous tests.

8.4 Introduce the mixture of air and sample in accordance with instructions of the instrument manufacturer. The flow rate should be moderate and reproducible. The pressure of gas in the oxidation chamber should be atmospheric and the temperature should be above the dew point of the gas being tested to prevent condensation of moisture on the oxidizing elements.

8.5 Read the percent combustible gas indicated on the meter. If the reading is low change to a lower range or change the orifice to one admitting less dilution air. If the instrument goes momentarily off scale on the high side and then returns to zero, the combustible gas content is probably above the upper explosive limit. This can be verified by additional dilution of the sample gas with air to bring the combustible gas content within the range of the instrument.

8.6 Recheck the instrument's zero balance and voltage adjustments.

8.7 Report reading in percent to the nearest small scale division.

8.8 Purge the instrument to remove sample gas in order to minimize corrosion and element contamination.

ELM:rw

MONS 204702

P. G. Benignus

GUIDE FOR THE DETECTION AND DETERMINATION OF GENERATED GASES IN OIL-IMMERSED TRANSFORMERS AND THEIR RELATION TO THE SERVICEABILITY OF THE EQUIPMENT

FIRST DRAFT - APRIL 10, 1972

PART I

Introduction

PART II

The Calibration and Use of Field Instruments for Detecting and Estimating the Amount of Combustible Gases Present in Gas Blankets Above Oil, Or in Gas Detector Relays

PART III

Procedures for Obtaining Samples of Gas and Oil from the Transformer for Laboratory Analysis

PART IV

Laboratory Methods for Analyzing the Gas Blanket and the Gases Extracted from the Oil

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Interpretation of Results in Terms of Transformer Serviceability

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PART I

The detection of certain gases generated in an oil-filled transformer in service is frequently the first available indication of a malfunction that may eventually lead to complete failure if not corrected. Arcing, corona discharge, low energy sparking, severe overloading, and overheating in the insulation system are some of the ways that can result in chemical decomposition of the insulating materials and the formation of various combustible and non-combustible gases. Normal operation may also result in the formation of some gases.

In a transformer, generated gases will be found dissolved in the insulating oil. They may also be found in the gas blanket above the oil or in gas collecting devices. The detection of an incipient fault, if present, involves an evaluation of the amount of generated gas present and the continuing rate of generation. An indication of the source of the gases and the kind of insulation involved can sometimes be gained by determining the composition of the generated gases.

This guide covers:

1. The calibration and use of field instruments for detecting and estimating the amount of combustible gases present in gas blankets above oil, or in gas detector relays.

2. Procedures for obtaining samples of gas and oil from the transformer for laboratory analysis.

3. Laboratory methods for analyzing the gas blanket and the gases extracted from the oil.

4. Interpretation of results in terms of transformer serviceability.

5. A bibliography.

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PART II

Arcing, corona discharge and localized overheating in the insulation system of transformers can result in chemical decomposition of the insulating materials and the formation of various gases. Detection of these gases is frequently the first available indication of a malfunction. Portable combustible gas meters are a convenient means of detecting the presence of generated gases.

1. Scope

1.1 This method covers the detection and estimation of combustible gases in the gas blanket above the oil or in gas detector relays in transformers using portable field instruments. Gases dissolved in the oil and non-combustible gases are not determined. A method of calibrating the instruments with a known gas mixture is included.

1.2 This method affords a rough quantitative estimate of the total combustible gases present in a gas mixture. If a more accurate determination of the total amount of combustible gases or a quantitative determination of the individual components is desired, a laboratory analytical method using a gas chromatograph or mass spectrometer should be used.

2. Summary of Method

2.1 A sample of gas is diluted with a fixed ratio of air and introduced into the meter at a pressure of approximately one atmosphere. Any combustible gases present are catalytically oxidized on the surface of an incandescent platinum alloy wire which is an element of a wheatstone bridge. When combustible gases oxidize on the surface they increase the temperature of the element which changes its resistance and upsets the balance of the bridge.

The change in the resistance of the indicating elements in the bridge circuit is indicated on a meter which is usually calibrated to read in per cent total combustible gas.

3. Apparatus

3.1 Portable combustible gas meter consisting of the following components:

3.1.1 Platinum alloy catalytic oxidizing elements incorporated in a wheatstone bridge circuit complete with a suitable battery and controls for balancing and voltage adjustment.

3.1.2 A milliampere meter preferably calibrated to read directly in per cent total combustible gases.

3.1.3 Gas mixing device for mixing air and sample gas at fixed ratios.

3.2 Standard reference gas.

3.2.1 A mixture of nitrogen and methane gas with a methane content in the mid-range of the instrument. The methane content should be known to the nearest 0.1 per cent.

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4. Safety Precautions

4.1 Gases generated in transformers can be violently explosive. The combustible gas meter should, therefore, be equipped with a flame arrester. Air should be used to dilute the sample gas. Pure oxygen should not be used for this purpose. Normal precautions should be observed when sampling the potentially explosive gases from the transformer.

5. General Considerations

5.1 In this method it is essential that sufficient oxygen be present in the gas mixture to oxidize the combustible gases. Since the gas blanket in a transformer is usually an inert gas, it is necessary to dilute the sample gas with a known amount of air. This is usually accomplished by either introducing air and the sample gas into the instrument in known ratios through fixed orifices, or by mixing known quantities of air and sample gas externally by displacement over water before introduction into the instrument. The working range of these instruments is between the low limit of sensitivity and the lower explosive limit. They generally read off scale at the high end between the lower explosive limit and the upper explosive limit, and indicate zero when the combustible gas content is above the upper explosive limit.

5.2 Contamination on the surface of the platinum alloy elements can seriously impair the sensitivity and response of the meter. This loss of response does not affect the normal balancing of the bridge circuit and is not detected by an electrical check of the instrument. It is, therefore, essential to check and calibrate the instrument frequently against a standard combustible gas mixture.

5.3 Since each combustible gas has its own individual instrument response, the values determined by an instrument are dependent upon the kind of combustible gas used for calibration. In order to permit interlaboratory comparisons of results, a mixture of methane in nitrogen has been selected as the standard calibrating gas as mixture.

6. Calibration

6.1 Prepare the instrument for operation and make zero balance and voltage adjustments in accordance with the instructions of the instrument manufacturer and the Procedure, Section 7.

6.2 Mix the standard reference gas with air and introduce it into the meter in exactly the same manner as used for the sample gas from the transformer.

6.3 Turn the calibration adjustment so the meter indicates the known methane content of the reference standard when the air and reference gas mixture is being tested. Purge the meter with air to remove traces of reference gas and proceed as in Section 7.

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PART II - (Cont'd)

7. Procedure

7.1 Prepare the instrument for operation in accordance with the instrument manufacturer's instructions. This should normally include a warm-up period to allow the battery voltage to stabilize, a voltage adjustment and an instrument bálance or zero adjustment.

7.2 Calibrate the instrument against the standard methane mixture following the procedure in 6. The frequency of calibration required for individual instruments can be determined by experience. Low readings because of contamination on the platinum elements can only be detected by a check with a gas of known composition. In any case of doubt the instrument should be recalibrated.

7.3 If the instrument has a range switch or more than one air dilution orifice, set for maximum range or dilution to avoid damage to the instrument. Purge the instrument so it is free of gas from previous tests.

7.4 Introduce the mixture of air and sample in accordance with instructions of the instrument manufacturer. The flow rate should be moderate and reproducible. The pressure of gas in the oxidation chamber should be atmospheric and the temperature should be above the dew point of the gas being tested to prevent condensation of moisture on the oxidizing elements.

7.5 Read the per cent combustible gas indicated on the meter. If the reading is low, change to a lower range or change the orifice to one admitting less dilution air. If the instrument goes momentarily off scale on the high side and then returns to zero, the combustible gas content is probably above the upper explosive limit. This can be verified by additional dilution of the sample gas with air to bring the combustible gas content within the range of the instrument.

7.6 Recheck the instrument's zero balance and voltage adjustments.

7.7 Report reading in per cent to the nearest small scale division.

7.8 Purge the instrument to remove sample gas in order to minimize corrosion and element contamination.

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PART III

3. Gas Samples for Laboratory Analysis

3.1 Gas Blanket - It has become common practice to extract samples of gas from the gas blanket above the oil in sealed transformers, for submission to the laboratory for quantitative analysis. This may be conveniently performed in accordance with the Tentative Method for Sampling Gas from a Transformer Under Positive Pressure, ASTM D2759-68T, being careful to obtain a representative sample.

3.2 Gas Dissolved in Oil - However, gaseous decomposition products of electrical insulating oil and solids are soluble in oil to varying degrees depending on their composition. It has therefore been recognized that the analysis of the gases dissolved in the oil provides a more sensitive procedure for detecting abnormalities in an incipient stage before serious damage has occurred.

In the case of oil-filled (diaphragm or conservator) transformers, until sufficient gas has accumulated to operate a gas detector relay, there is no gas in the transformer tank above the oil; hence, the only gas available for analysis is that dissolved in the oil.

3.2.1 Apparatus - The sampling device consists of a precision ground glass syringe terminated with a Luer-Lok fitting to which is attached a plastic three-way stopcock (syringe stopcock). This stopcock, although removable, is to be regarded as an integral part of the sampling device and any subsequent references to the syringe imply the presence of this stopcock. A second identical stopcock which does not stay with the syringe, is used in some cases and will be referred to as the auxiliary sampling stopcock.

Before sampling is commenced, all syringes should be examined for the absence of dirt or grit and the syringe piston should be lubricated with a thin film of clean transformer oil and checked for smooth sliding. The syringe stopcock should be checked for tightness in the Luer-Lok fitting but must not be overtightened to avoid stripping the flange.

Auxiliary Fittings - For taking oil samples, an adapter must be made which can be fitted to the transformer valve and which will accept the tapered inlet of the auxiliary sampling stopcock. (This has a standard Luer taper, minimum diameter 5/32", maximum diameter 11/64", taper length 5/16"). A Luer taper can be bored in the transformer valve plug or, alternatively, a female Luer hub (cut down from a large, e.g. 15, gauge hypodermic needle) can be soldered into a suitably sized hole drilled in the transformer valve plug (Figure 1). Suitable needles can be obtained from medical or laboratory supply houses.

Alternatively, a hose nipple may be fitted to the transformer valve to accept one end of the plastic tubing of the connecting

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PART III - (Cont'd)

3.2.1 Auxiliary Fittings - (Cont'd)

device shown in Figure 3. This may be made using 1/4" NPS (6.4 mm) copper tubing provided with soldered knurls or beads near each end to accept the plastic tubing, to which, using a T connection near the center, the (metal) Luer-Lok auxiliary sampling stopcock is soldered. The rubber tubing should be 1/4" I.D. by 1/16" wall, and the spring fitting made to suite. This alternate arrangement has been found convenient when the desired sampling transformer valve location is difficult to reach. The spring loading of the syringe has been found useful but not indispensable.

3.2.2 Oil Sampling - A satisfactory technique for taking bubble-free oil samples is shown diagramatically in Figures 1 and 2. The flushing ports of the syringe and auxiliary stopcocks are shown horizontal in Figure 1, but in practice it is better if they are directed downward to facilitate collection of the waste oil. (NOTE: The handles of the plastic stopcocks always point to the closed port leaving the other two ports in open communication.)

The technique described requires the oil to be under a positive pressure (0.2 psig or greater). All stopcock switching should be smooth and fairly rapid to minimize pressure build-up during the momentary cut-off between positions. It should be noted that a continuous oil flow is maintained throughout the entire procedure.

a) Attach the adapter plug to the transformer valve and flush until all trapped air is eliminated and then adjust the oil flow to approximately 50-100 mls/min. With its handle in the flush position, plug in the auxiliary sampling stopcock and check to ensure that a smooth, bubble-free flow is obtained from the flushing port. (Figure 2.1).

b) With its stopcock in the fill position, attach the syringe to the auxiliary sampling stopcock and turn the auxiliary sampling stopcock to the fill position (Figure 2.2). Allow 5-10 mls of oil to enter the syringe and then return the auxiliary stopcock to its flush position and separate the syringe (Figure 2.3).

c) With the syringe vertical eject any air bubbles (Figure 2.3A) and then depress the piston to the zero mark and close the syringe stopcock (Figure 2.3B).

d) The syringe, bubble-free and with its dead volume filled with oil, is then reattached to the auxiliary sampling stockcock. The auxiliary stopcock is turned to the fill position and the oil stream then flows from the flushing port of the syringe stopcock (Figure 2.4).

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PART III - (Cont'd)

3.2.2 Oil Sampling - (Cont'd)

e) Turn the syringe stopcock to the fill position and allow oil pressure to push the piston back until the syringe is filled with oil (Figure 2.5). Do not pull the piston manually since this can result in bubble formation.

f) Return the syringe stopcock and then the auxiliary stopcock to their flush positions (Figure 2.6) and separate syringe.

NOTE
Bubbles may form in the syringe subsequent to sampling but, provided the sample was bubble-free initially, this will not affect the subsequent analysis and under no circumstances should attempts be made to remove such bubbles.

If it is necessary to obtain a sample of oil from a difficult location using the alternate auxiliary connecting tubing, attach one end of the plastic tubing of the sampling device to the hose nipple and mount the syringe vertically on the copper tubing. With both stopcocks closed, open the valve slowly and allow the oil to flow freely through the tubing. Open both stopcocks and at the same time close or pinch the free end of the plastic tubing. This will permit the oil to enter the syringe and force the plunger upwards. Remove the plunger and let the oil overflow the barrel, at the same time wetting the plunger completely. Insert the plunger back into the barrel taking care not to trap air. At the same time open the pinched end and apply pressure on the plunger to remove the oil from the syringe. Repeat this procedure twice more but without removing the plunger from the syringe.

When the syringe is filled for the third time close both stopcocks, open the pinched end and close the valve. Inspect the oil sample for absence of air bubbles and attach the spring device to the syringe.

3.2.3 Shipment - All syringes should be wiped clean, identified and placed in individual syringe cartons. Six of these cartons fit into a shipping container (if less than six samples are sent the container must be filled out with empty cartons). To minimize delay between sampling and analysis, samples should be shipped to the laboratory by the most rapid method. They may be shipped by first class mail or air mail if distances warrant.

NOTES
Medical syringe, Becton Dickinson B-D Yale 2314, 30 ml or 100 ml capacity has been found suitable.

Tomac three-way stopcock, Cat. No. 17108-010 K-75 from American Hospital Supply Company, Evanston, Illinois 60201, has been found suitable.

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PART III - (Cont'd)

3.2.3 (Cont'd)

NOTES
Luer-Lok metal stopcock, Model No. 3135 (MS01) Spring Clip or No. 3136 (MS01-T) Screw Type, from Becton Dickinson and Company, Rutherford, New Jersey, has been found suitable.

Tygon plastic tubing from U. S. Stoneware, Inc. has been found suitable.

Syringe and shipping cartons for handling of 30 ml syringes containing oil samples for gas or water analysis may be ordered as follows:

Individual syringe carton Q12012;
Cat. No. 112329, S69-3168
For 30 ml syringe, complete with insert.

Master shipping carton Q12012;
Cat. No. 116823, S69-3341
For 6 syringe cartons.

Manufacturer: Consolidated Bathurst Packaging, Ltd.
1035 Hodge Street
St. Laurent, Quebec

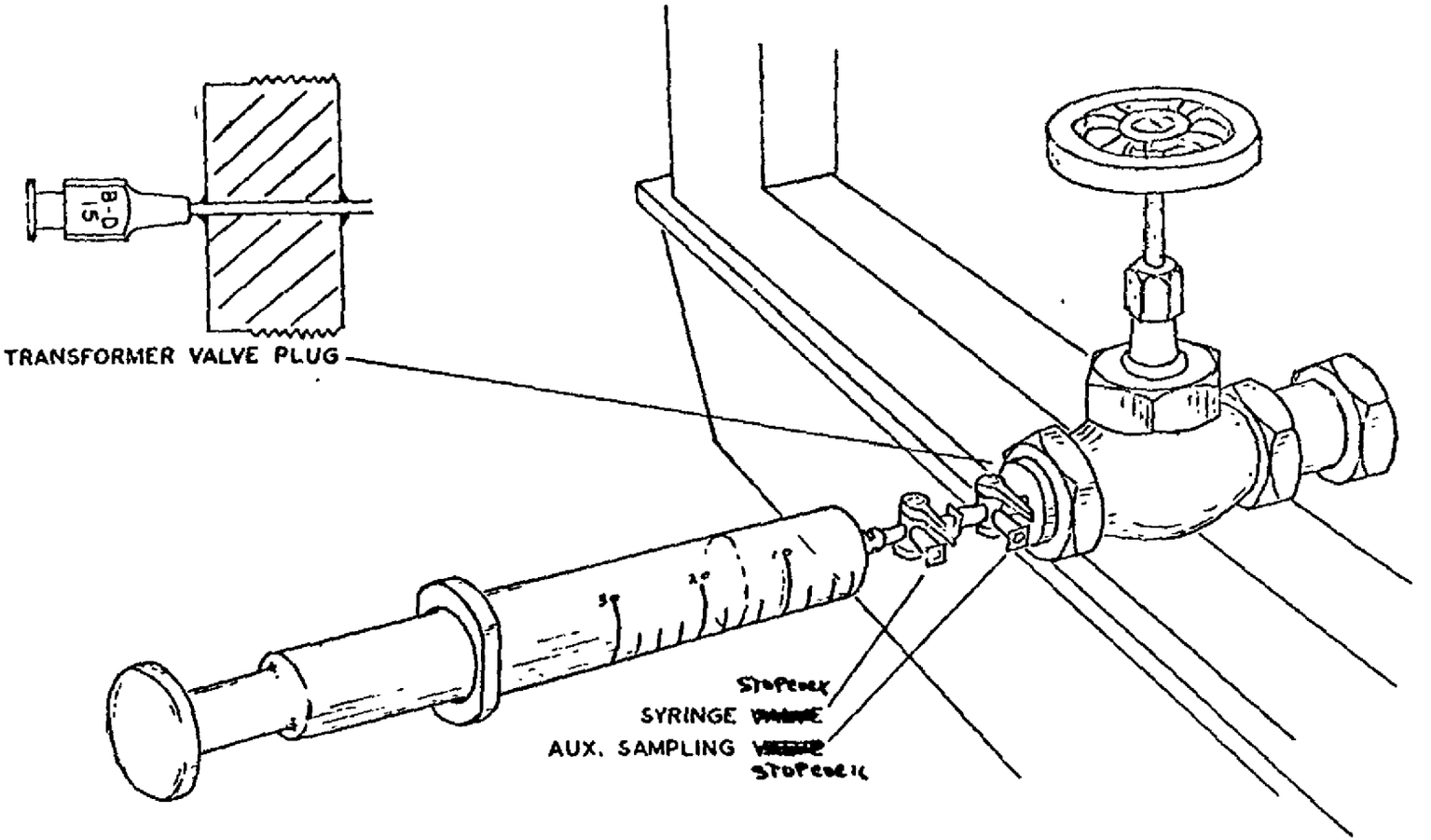

FIGURE 1

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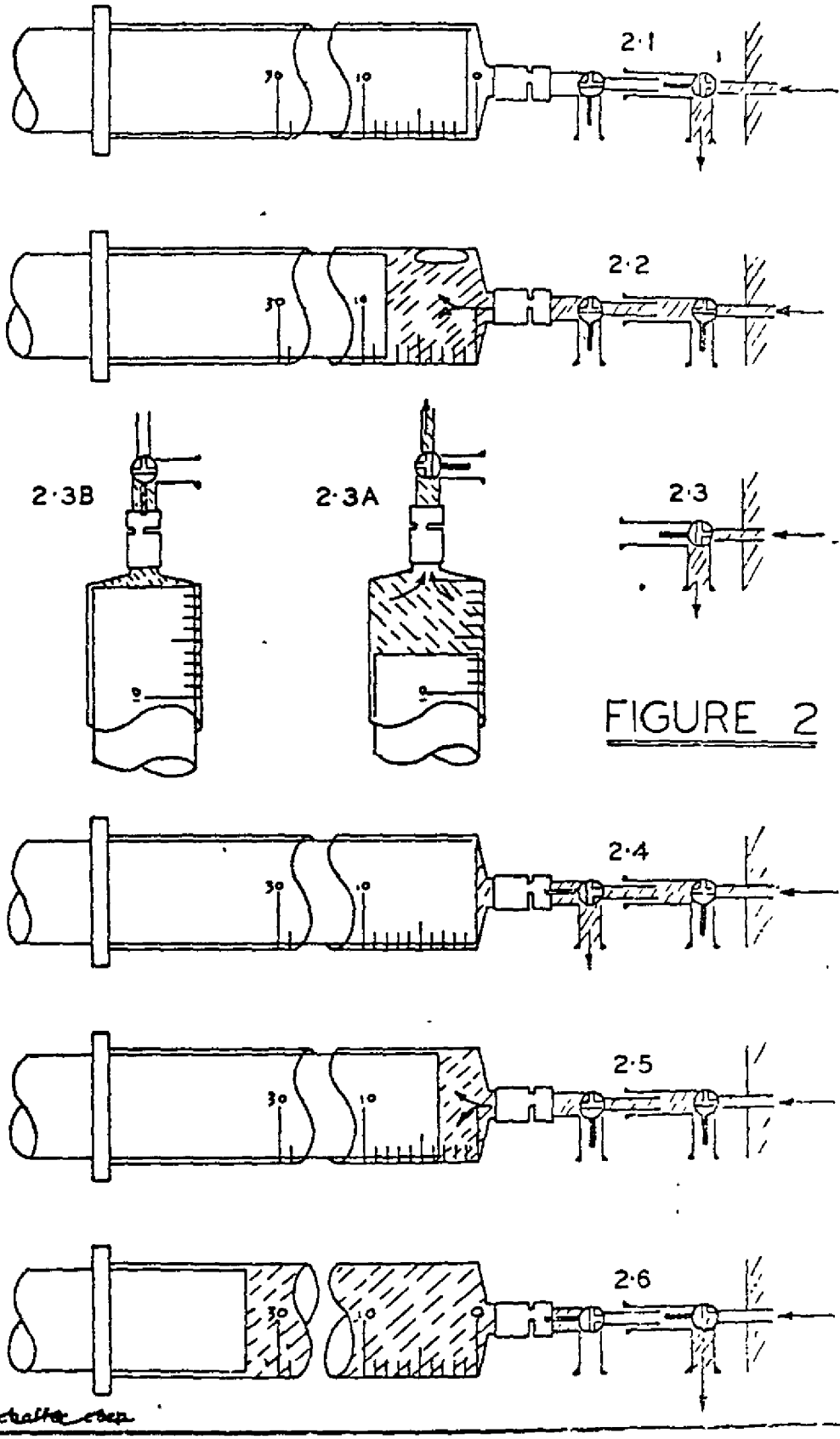

FIGURE 2

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SYRINGE-TYPE DEVICE

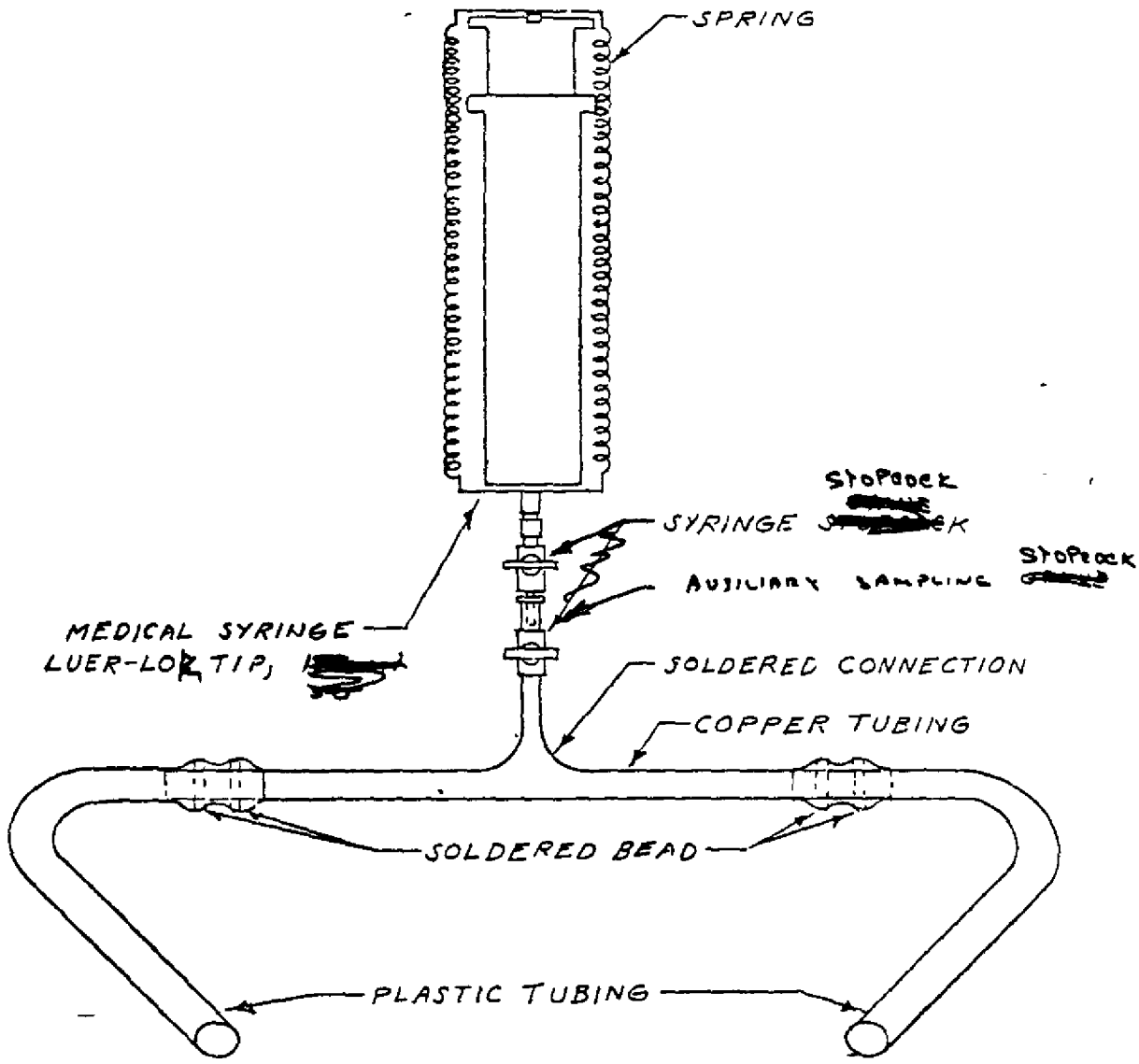


FIGURE 3

MONS 204714

PART IV

4 Gas Extraction and Analysis

4.1 Gas Blanket Sample

The sample of the gas blanket may be submitted directly to analysis as described in section 4.3.

4.2 Gas Extraction from Oil Sample

4.2.1 Extraction of Non-Acidic Gases by Purging with Carbon Dioxide.

4.2.1.1 Scope

4.2.1.1.1 This method covers the extraction of gases present in insulating liquids with a Saybolt Universal viscosity of 100 or less at 100 C, for quantitative analysis. Any gas that is nonreactive with a strong caustic solution may be determined; carbon dioxide cannot be determined.

4.2.1.2 Summary of Method

4.2.1.2.1 The method consists essentially of purging dissolved gasses from a liquid sample with pure carbon dioxide gas and carrying them into a graduated buret filled with a potassium hydroxide solution for measurement and determination. The carbon dioxide is completely absorbed by the potassium hydroxide solution and the volume of the other gases is measured. The extracted gas is then diluted with a helium to a volume suitable for injection into a gas chromatograph.

4.2.1.3 Description of Terms

4.2.1.3.1 Gas Content by Volume of an insulating liquid is the total volume of gas contained in a given volume of liquid. It is expressed as a percentage measured at 760 TORR and 0°C.

4.2.1.3.2 Volume Percent of Gas is the percentage of an individual gas at 760 TORR and 0°C contained in a given volume of liquid.

based on the original volume of liquid from which the gas was extracted.

4.2.1.4 Apparatus

4.2.1.4.1 The apparatus, shown in Figure 1, consists of the following components:

4.2.1.4.2 A buret with a calibrated capacity of 25 ml and calibrated in 0.1-ml divisions. The buret shall be provided with a liquid levelingbottle of suitable capacity[1], connected with plastic tubing.[2]

4.2.1.4.3 A Pregle-type micro stopcock to allow delivery of gas to the buret or venting the gas to the atmosphere.

4.2.1.4.4 A sample purging chamber[3] having approximately 130 ml capacity and fitted with a medium porosity disc. A flexible heating tape, wrapped around the purging chamber is necessary, when samples are to be purged at temperatures in excess of room temperature.

4.2.1.4.5 A sample delivery tube, made of a length of 1/16 inch (1.6mm) inside diameter stainless steel tubing, tightly inserted in

(1) A polyethylene aspirator bottle, 16 ounces capacity, Bel-Art Products, Pequannock, N.J. 07440, Part No. H-11880, has been found satisfactory.

(2) Tygon plastic tubing available from Norton Plastics and Synthetics Division, has been found satisfactory.

(3) An absorption tube, Corning No. 39620 modified to contain a medium porosity disc, has been found to be satisfactory.

one of the holes in the stopper, and extending to the fritted glass filter. A syringe stopcock[4] shall be soldered to the tube to permit delivery of an accurate volume of liquid, to subsequently seal the purging chamber and then to facilitate removal of the extracted liquid sample from the purging chamber.

4.2.1.4.6 A gas flowmeter[5], having a range from 0 to 80 cc/min of air, to maintain a pre-set and constant flow of carbon dioxide.

4.2.1.4.7 An elbow adapter, consisting of a 12/2 ball joint and having a drawn-down tip suitable for insertion in a small hole in the rubber stopper.

4.2.1.4.8 A stopper of oil-resistant rubber, to fit the sample-purging chamber and provided with two small holes to allow tight fitting of the elbow adapter and sample delivery tube.

4.2.1.4.9 A glass capillary connecting tube, 2 mm i.d., suitably bent and with ball and/or socket joints to provide a connection from the flowmeter to the sample-purging chamber.

(4) A stopcock, female Luer-Lok tip to male Luer slip, available from Becton, Dickinson and Company, Rutherford, New Jersey, Model No. 3135 (MS01) Spring Clip, has been found to be satisfactory.

(5) A flowmeter (rotameter), Scientific Glass Apparatus Co. Inc., Bloomfield, N.J., No. JG-6110, Tube No. 0232-150, has been found satisfactory.

4.2.1.4.10 Ball-and-socket clamps, size 12, for tightly securing all joints to prevent leakage.

4.2.1.4.11 A sampling device consisting of a calibrated glass medical syringe fitted with a syringe stopcock[4] and a spring device for sealing of the sample during transfer or storage. The syringe shall have a calibrated capacity of 100 ml and shall be capable of accurately delivering a liquid volume within the accuracy required for the method.

4.2.1.4.12 A cylinder containing pure compressed carbon dioxide[6] fitted with a two stage regulator, permitting control over an unvarying delivery pressure.

4.2.1.4.13 A metering valve[7] to regulate the flow of carbon dioxide as indicated by the flowmeter.

4.2.1.4.14 A suitable length of 1/8 inch copper tubing to connect from the carbon dioxide cylinder to the metering valve.

4.2.1.4.15 A precision nitrometer (azotometer) as described in ASTM standard method D 1827, paragraph 5(a). It is used to determine the gas blank and is not shown in Figure 1.

4.2.1.5 Reagents

4.2.1.5.1 Purity of Reagents - Reagent grade chemicals shall be used in

(6) Coleman Instruments Grade, available from The Matheson Company, Inc., East Rutherford, N.J., with a purity of 99.99 vol. % minimum, has been found suitable.

(7) A Milli-Mite forged needle valve, Hoke No. 1315-M4B, without micrometer attachment, has been found satisfactory.

all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

4.2.1.5.2 Purity of Water - Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to the Specifications for Reagent Water (ASTM Designation: D 1193).

4.2.1.5.3 Cylinder of Carbon Dioxide.[6]

4.2.1.5.4 Mercury

4.2.1.5.5 Potassium Hydroxide Solution (40 percent) - Dissolve 572 g of potassium hydroxide (KOH) in water and dilute to 1 liter.

4.2.1.5.6 Stopcock Grease that is resistant to KOH and the oil sample.

4.2.1.5.7 Cylinder of Helium.[8]

4.2.1.6 Preparation of Apparatus

4.2.1.6.1 Assemble apparatus as shown in Figure 1.

4.2.1.6.2 Fill the bottom portion of the buret with mercury to a point about 5 to 7 mm above the gas entry capillary. The mercury acts as a valve to allow the gas to bubble into the buret and also prevents the KOH solution from flowing into the system due to gravity.

4.2.1.6.3 Open the buret stopcock and add KOH solution to the leveling bottle until the buret is completely filled and free of air bubbles.

4.2.1.6.4 Prepare the precision nitrometer, used for the determination

(8) Reactor Grade Helium with a purity of 99.996 volume % minimum.

of the gas blank, similarly to method described in paragraph 7.2.

4.2.1.7 Procedure

4.2.1.7.1 Determination of gas blank. Connect the micro stopcock to the precision nitrometer. Allow the carbon dioxide to flow through the purging chamber, to exit first through the sample delivery tube and after closing the stopcock at the delivery tube through the vent of the micro stopcock to remove all air. Then adjust the micro stopcock to allow the carbon dioxide to flow into the precision nitrometer. Adjust the flow rate (Note 1) and allow the carbon dioxide to flow until all air has been displaced from the side arm. This is indicated when gas bubbles rapidly decrease to micro size near the top of the KOH solution. Expel the trapped air through the stopcock on top of the nitrometer, close the stopcock, lower leveling bottle somewhat and simultaneously start a timer. At the end of the timed period turn the micro stopcock in order to exhaust the carbon dioxide to the atmosphere. Then hold the liquid in the leveling bottle at the level of the liquid in the nitrometer column, and read the volume of the gas collected. Record the volume, time and flow rate used.

4.2.1.7.2 Preparation for the extraction. Disconnect the precision nitrometer from the micro stopcock, connect the buret and purge system as in paragraph 8.1. Check flow rate of carbon dioxide gas, start a timer and lower leveling bottle somewhat.

NOTE 1: Maintain the same flow rate and time for the determination of the blank and for the extraction of the sample.

A rate of about 30 ml per minute and a 15 to 20 minute period have been found to be satisfactory. Several determinations of the blank should be made to assure complete removal of air from the system. Once a value for the blank has been established, it is not necessary to determine it for subsequent extractions of insulating liquids provided the same carbon dioxide cylinder is used and time and flow rate are not varied from the original determination.

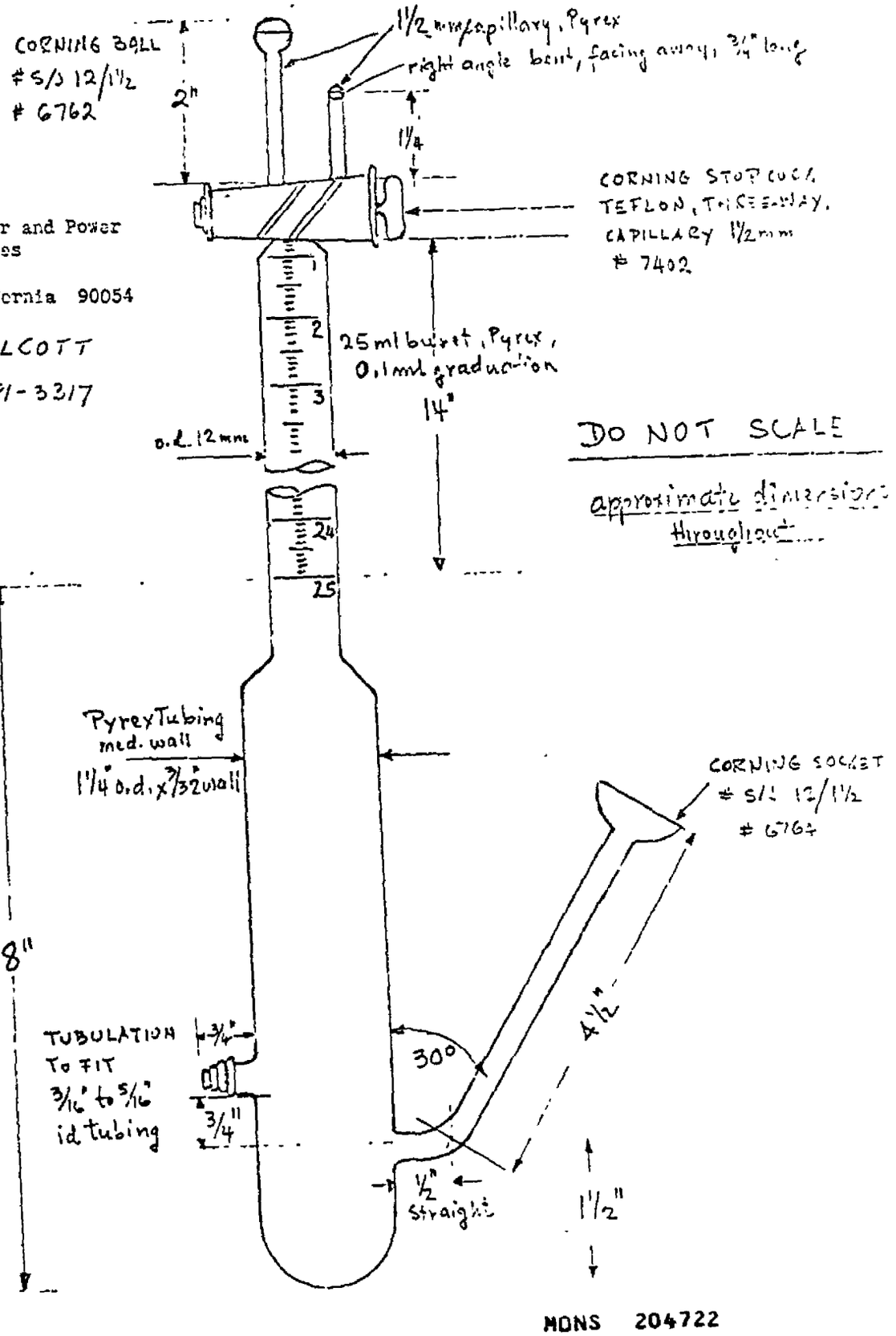
4.2.1.7.3 Extraction of the sample. Introduce a measured amount of oil slowly from the syringe through the stopcock on top of the sampling tube, making sure that no air enters at the same time. This can conveniently be done by filling the barrel of the stopcock with a small amount of oil. The extraction can be terminated when the gas bubbles again become micro size. Stop the timer and vent the gas at the micro stopcock to the atmosphere.

4.2.1.7.4 Determination of volume of extracted gas. After allowing sufficient time for drainage of the buret, hold the liquid in the leveling bulb at the same level as the liquid in the buret, read and record the volume of gas collected. From this amount deduct the value for the blank previously obtained, using the same length of time and gas flow rate.

4.2.1.7.5 Convert the measured volume of extracted gas and convert it to volume at 760 Torr and 0°C, and record it as a percentage, as the Total Gas Content by Volume.

GAS COLLECTING BURET

Front View



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approximate dimensions throughout

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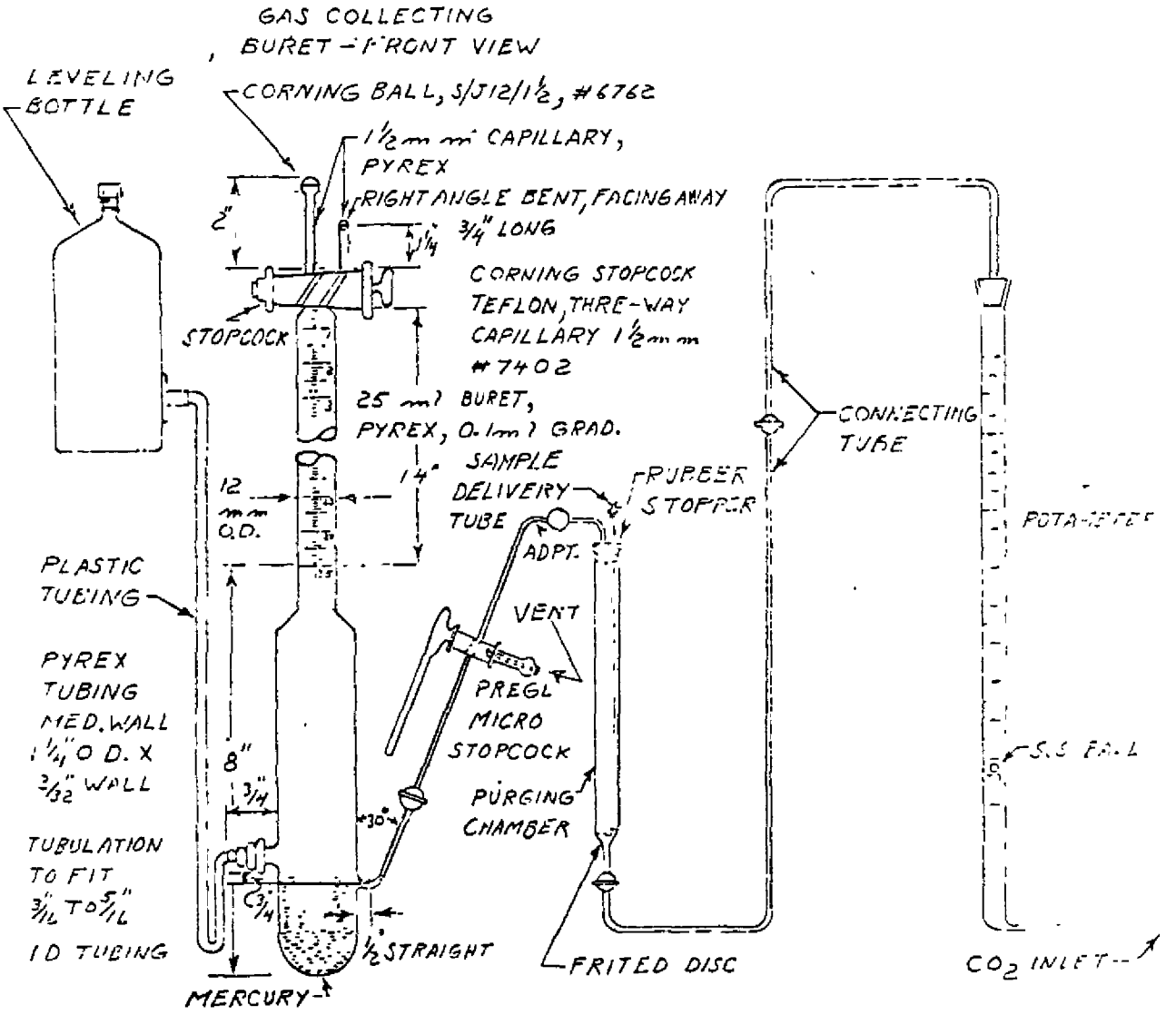

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NOT TO SCALE

4.2.2 Extraction of Gases by Expansion in an Evacuated Flask

4.2.2.1 Scope

4.2.2.1.1 This method covers the extraction of gases present in insulating liquids with Saybolt Universal viscosities of 100 or less at 100°C, for quantitative analysis. Any gas may be determined.

4.2.2.2 Summary of Method

4.2.2.2.1 The method consists essentially of extracting dissolved gases from a liquid sample by expansion into a pre-evacuated known volume. A known portion of the extracted gas is then compressed, measured and contained for transfer to the analytical instrument.

4.2.2.3 Description of Terms

4.2.2.3.1 Gas Content by Volume of an insulating liquid is the total volume of gas contained in a given volume of liquid, expressed as a percentage at 760 Torr and 0°C.

4.2.2.3.2 Volume Percent of Gases is the percentage of individual gases, at 760 Torr and 0°C contained in a given volume of liquid, based on the original volume of liquid from which the gas was extracted.

4.2.2.4 Apparatus

4.2.2.4.1 The apparatus, shown in Figure 1 is suitable for use with a 30 ml sample of oil and consists of the following components.

4.2.2.4.2 A narrow bore teflon tubing terminated with a Luer fitting to accept a glass syringe and leading to a solid plug, three way high vacuum stopcock (B) lubricated with high vacuum silicone grease.

4.2.2.4.3 A 100 ml. degassing flask which may be evacuated through a vacuum trap containing a Teflon coated magnetic spin bar, and mounted on a magnetic stirrer.

4.2.2.4.4 A vacuum pumping system capable of at least 10^{-6} Torr at blank off and capable of evacuating the system to 10^{-5} Torr. This will usually consist of a diffusion pump and a back-up pump.

4.2.2.4.5 Glassware consisting essentially of a large volume McLeod gauge, sufficiently large compared with the gas content of the sample so that virtually complete degassing is obtained, and so that the collection ratio shall be as large as possible. The dimensions shown in Figure 1 will provide for approximately 99% degassing of a 30 ml sample of oil, and a collection ratio of approximately 80%.

4.2.2.4.6 Stopcocks 1 to 5 are greaseless high vacuum stopcocks with Teflon plugs. Stopcocks A and B are solid plug, three way, high vacuum stopcocks lubricated with high vacuum silicone grease.

4.2.2.4.7 A septum retainer sealed to the end of the calibrated compressed gas collection tube, fitted with a silicone rubber septum.

4.2.2.5 Preparation of Apparatus

The apparatus shall be carefully checked for vacuum tightness of all joints and stopcocks. The volume of the total expansion space and the volume of the gas trapped in the collection tube shall be carefully measured and the volumetric collection ratio calculated. The gauge constant of

the collection tube shall be calculated, and the tube clearly calibrated.

4.2.2.6 Procedure

4.2.2.6.1 Attach the sampling syringe to the teflon delivery tube connected to the degassing flask through stopcock B. Open the syringe valve stopcock and force a small amount of oil into the flask to fill the teflon tubing and the bore of the stopcock. Turn stopcock B to connect to the other limb and adjust the volume of oil in the sampling syringe to exactly 30 mls., by forcing excess oil into the overflow breaker. If the sampling syringe contains free gas bubbles, care must be exercised to ensure that they are retained in the syringe with the oil sample. Close stopcock B.

4.2.2.6.2 Clamp the sampling syringe in a vertical position to ensure that any free gas bubbles will be forced into the degassing flask with the oil sample.

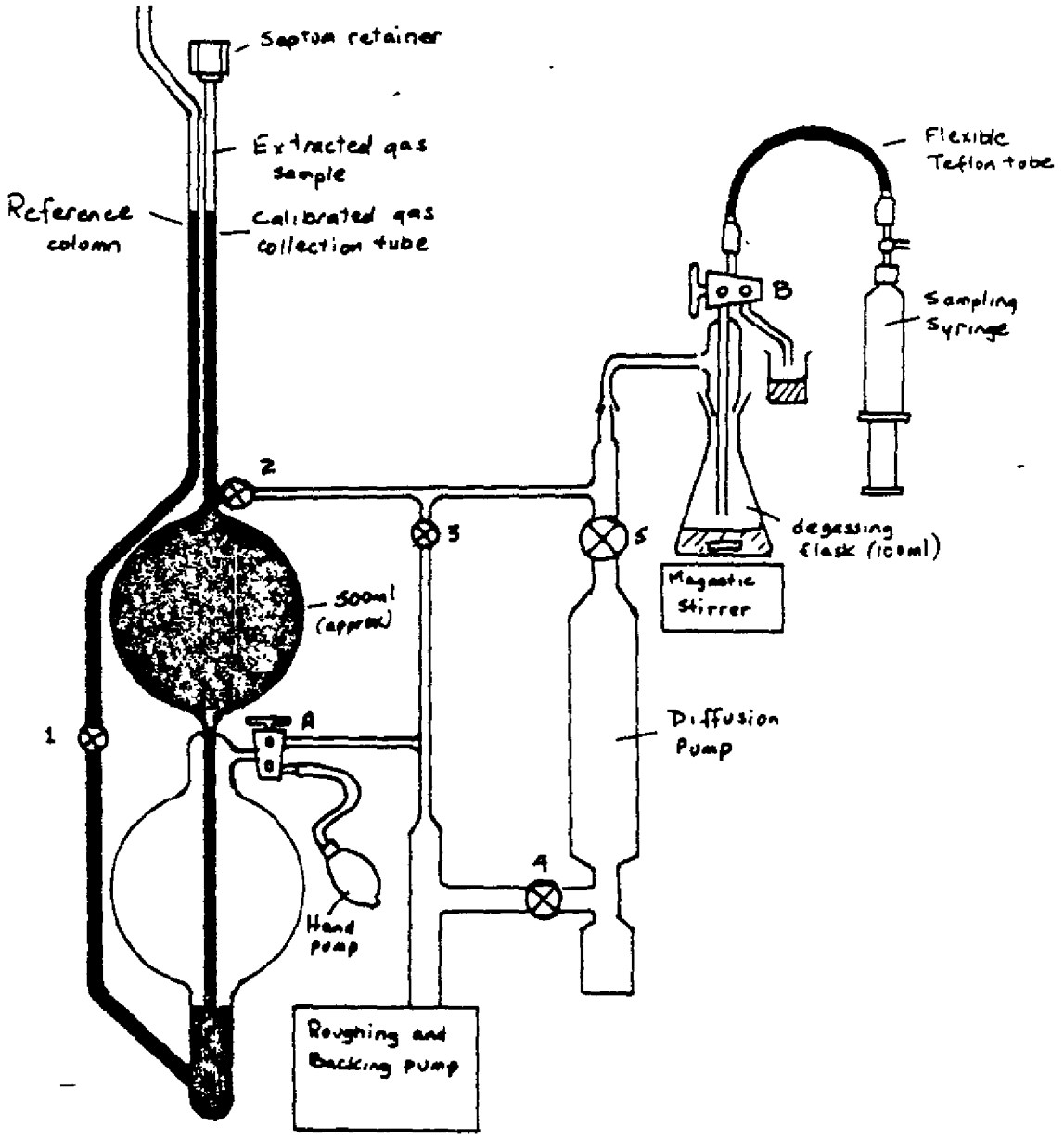
4.2.2.6.3 Evacuate the complete system as far as stopcock B to an absolute pressure of approximately 10^{-5} Torr. Isolate the expansion space from the vacuum pumps.

4.2.2.6.4 Slowly open stopcock B to allow the oil sample to enter the degassing flask, while exercising care that the syringe piston is free to move into the syringe barrel. Turn on the magnetic stirrer to provide vigorous agitation of the oil sample. Transfer exactly 30 mls. of oil into the degassing flask and close stopcock B.

4.2.2.6.5 Isolate the predetermined fraction of extracted gas in the collection tube and compress it to atmospheric pressure by

increasing the pressure in the lower flask and permitting the mercury column to rise up in the collection tube to a level equivalent to that in the reference column.

4.2.2.6.6 Measure the volume of extracted gas and convert it to volume at 760 Torr and 0°C and record it as a percentage, as the Total Gas Content by Volume.


Septum retainer
Extracted gas sample
Reference column
Calibrated gas collection tube
Flexible Teflon tube
B
Sampling Syringe
2
3
5
degassing flask (100ml)
Magnetic Stirrer
500ml (approx)
1
A
Diffusion Pump
4
Hand pump
Roughing and Backing pump

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PART IV - (Cont'd)

4.3 Determination of Individual Gases by Gas Chromatography

4.3.1 Scope

4.3.1.1 This method covers the determination of the individual gases in a composite gas sample. It is applicable to the analysis of hydrogen, methane, ethane, ethylene, acetylene, propane, propylene, nitrogen, oxygen, carbon monoxide, and carbon dioxide.

4.3.2 Significance

4.3.2.1 Oil impregnated electrical insulating materials may decompose under the influence of thermal and electrical stresses, and in doing so generate gaseous decomposition products of various composition. The nature and amount of the individual component gases that may be recovered and analyzed may be indicative of the type and degree of the abnormality responsible for the gas generation.

4.3.3 Summary of Method

4.3.3.1 The gas sample is introduced into a series of gas chromatographic columns that will separate the individual constituent gases. The composition of the sample is calculated from its chromatogram by comparing the area of the peak of each component with the area of the peak of the same component on a reference chromatogram made on a standard mixture of known composition.

4.3.4 Apparatus

4.3.4.1 A Gas Chromatograph equipped with a thermal conductivity detector or equivalent in sensitivity and stability, and at least two adsorption columns capable of separating the component gases

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in the ranges of volume percent concentration shown in Table I.

TABLE I

	Content by Volume in the Gas Sample	
	Minimum	Maximum
Hydrogen	0.05	95
Methane	0.003	100
Ethane	0.002	100
Ethylene	0.01	100
Acetylene	0.01	100
Propane	0.01	100
Propylene	0.01	100
Nitrogen	0.005	100
Oxygen	0.005	100
Carbon Monoxide	0.005	100
Carbon Dioxide	0.005	100

4.3.4.2 Any suitable adsorbent may be used. Molecular Sieves, Porapak Q, Porapak S, Hexamethylphosphoramide (HMPA), Diisodecyl phthallate A, Silica Gel J, and Chromosorb 102 have been found suitable.

4.3.5 Reagents and Materials

4.3.5.1 A cylinder of Helium Gas.

4.3.5.2 A reference standard gas mixture containing known percentages of the gases shown in Table I.

4.3.6 Calibration

4.3.6.1 Prepare the gas chromatograph for use as directed by the manufacturer, and establish a set of operating conditions capable

of complete separation of the indicated component gases. The following conditions have been found suitable:

Carrier gas	Helium
Flow rate	Molecular Sieve column 85 ml/min HMPA column 35 ml/min
Column Oven Temperature	25°C
Injection Port Temperature	25°C
Detector Temperature	
Sample Volume	Approximately 2 ml.

4.3.6.2 Inject a known volume of the reference standard gas mixture on the adsorption columns being used and establish a pattern of elution times for the gas components known to be in the mixture, at an established set of operating conditions and sample size. Repeat the analyses until consistent operating conditions provide consistent chromatograms. Calibration should be repeated daily when analyses are being conducted.

4.3.7 Procedure

4.3.7.1 Inject the established volume of the unkown gas mixture on the adsorption columns in strict accordance with the conditions established for calibration. Repeat the analyses until consistent chromatograms are obtained.

4.3.8 Calculation

4.3.8.1 Measure the height, and the width at half height of each of the peaks of the chromatogram, and calculate the area of each peak:

Peak Area = Height x Width (at half height)

4.3.8.2 Identify the gases represented by each peak by comparison of

elution times with those obtained for the reference standard gas mixture in the calibration procedure.

4.3.8.3 Determine the amount of each identified gas component by comparing respective peak areas with those obtained for the reference standard gas mixture in the calibration procedure.

4.3.8.4 Calculate the volume concentration of each identified gas component in the gas sample, as the percentage of the volume of the individual gas with respect to the total volume of the gas sample used, and express this as the Content by Volume (of the individual gas).

4.3.8.5 If the gas sample used in the analysis had originally been extracted from a sample of oil, calculate the volume concentration of each identified gas component at 760 Torr and 0°C, as the percentage of the volume of the individual gas with respect to the volume of the original oil sample, and express this as the Volume Percent (of the individual gas) In Oil at 760 Torr and 0°C. The probable range of reported values are shown in Table II.

TABLE II

	Volume Percent in the Oil Sample	
	Minimum	Maximum
Hydrogen	0.001	5
Methane	0.0001	10
Ethane	0.0002	10
Ehtylene	0.0002	10
Acetylene	0.0002	10

PART IV - (Cont'd)

	Minimum	Maximum
Propane	0.0005	10
Propylene	0.0005	10
Nitrogen	0.0002	10
Oxygen	0.0002	10
Carbon monoxide	0.0002	10
Carbon dioxide	0.0002	10

PART V

Gas samples used to interpret the serviceability of transformers in this guide are obtained by any of the following three methods.

1. A sample from the gas space above the oil in the transformer.
2. A sample of the gas extracted from a representative portion of the oil in the transformer.
3. A sample of the gas collected in a gas relay such as a Buckholtz relay.

It is important to understand in this type of analytical work that the actual percentages of the gas components will differ widely depending upon the method of obtaining the sample as indicated above even though the gas components are formed at the same source. This is basically due to differences in the solubility of the various gases in transformer oil. The following values show the oil solubility of different gases at 760 mm Hg. and temperatures of 25°C and 75°C.

	25C	75C
Hydrogen (H_2)	7% by volume	
Nitrogen (N2)	8.6% by volume	
Carbon monoxide (Co)	9% by volume	
Argon (Ar)	15 % by volume	
Oxygen (O_2)	16% by volume	
Methane (CH_4)	30% by volume	
Carbon dioxide (CO_2)	120% by volume	
Ethane (C_2H6)	280% by volume	
Ethylene (C_2H_4)	280% by volume	
Acetylene (C_2H_2)	400% by volume	

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PART V (Cont'd)

Propylene (C_3H_6)	1200% by volume
Propane (C_3H_8)	1900% by volume

The solubility of air in oil, allowing for the different partial pressures and solubilities of the individual components, nitrogen, oxygen, argon and carbon dioxide, is approximately 10.3%. The composition of the constituents in oil by analysis is approximately 70% nitrogen; 30% oxygen and argon; and 0.3% carbon dioxide by volume.

When a fault occurs in a transformer the newly formed gases partly dissolve in the oil causing some of the oxygen and nitrogen to leave the oil. This exchange continues until a new state of equilibrium is attained, this being determined by the partial pressure of the individual gases and their solubilities. Thus the gases in the gas space are high in the low oil soluble gases and the gases in the oil are high in the more oil soluble gases.

Likewise some changes in the gas composition in transformers can take place do to the chemical reaction of the gases with the oil, such as the reaction of oxygen, hydrogen or acetylene and the oil. It can be seen that the interpretation of gas analyses is difficult.

Conditions causing gas formation in Transformers.

1. High energy arcing under oil with or without cellulose. The gases produced contain large percentages of hydrogen and acetylene with lesser amounts of methane and ethylene. If the arc is associated with cellulose significant amounts of carbon dioxide and carbon monoxide are formed. If the fault is not in a cellulose area the carbon oxides are not present. The volume of gas generated is very high.

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PART V (Cont'd)

2. Low energy spark discharges. The gases produced are the result of low temperature breakdown of oil and consist primarily of hydrogen and some methane with a small amount of ethane. Acetylene is not present nor are carbon dioxide or carbon monoxide. This type of fault only produces small amounts of gases but this can continue over an extended period usually without failure of the transformer.

3. Severe local overheating with or without cellulose. This type of fault is usually caused by a turn-to-turn failure or a poor brazed or other type connections. The fault causes a high temperature breakdown of oil resulting in high hydrogen and about equal amounts of methane and ethylene. In some cases small amounts of acetylene can be detected. Carbon dioxide and carbon monoxide are found where cellulose is present.

4. Carona discharge in oil. Corona discharges can cause oil breakdown and may occur in voids and other gas pockets. It is characterized by the formation of significant amounts of hydrogen with only traces of methane and ethylene.

5. Thermal decomposition of cellulose. This can result from overheating of transformer insulation by overloading equipment or in cases where normal oil circulation is impaired and high temperature result in the transformers. The gases produced are primarily carbon dioxide and carbon monoxide with occasionally some hydrogen. In extreme cases the low molecular weight hydrocarbons can result from oil breakdown.

6. Rusting and dissociation of water. In some cases significant quantities of hydrogen are formed from excessive moisture in critical areas of the transformers. The moisture can result in rusting of the core and dissociation to produce hydrogen and oxygen. Other combustible gases are not found.

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Classification of Transformers based on Gas Analyses by Combustible Gas Meter.

1. 0 - .5% Combustible gases.

Very low percentages of non combustible gases permits continued operation of equipment with no planned monitoring procedure.

2. 0.5% - 2% Combustible gases.

Low percentages of gases permits continued operation with periodic monitoring procedures using a combustible gas meter to establish the rate of production of the gases and if further gas analysis are required.

3. 2 - 5% Combustible gases.

Intermediate quantities of combustible gases requires gas analysis, purging, monitoring and internal inspection at earliest opportunity to determine possible causes and solution to the formation of combustible gases without transformer teardown.

4. Over 5% Combustible gases.

High quantities of combustible gases requires gas analysis, removal of equipment from service, careful purging, immediate internal inspection with transformer dismantling if the obvious causes are not corrected by the internal inspection and changes.

Interpretation of Gas Analysis

The following table shows the results of gas analyses and action that is suggested by the types of gases found.

INTERPRETATION OF GAS ANALYSIS

	Results of Gas Analysis	Action to be taken on Transformer.
1.	Nitrogen plus 5% or less oxygen	Normal operation of transformer.
2.	Nitrogen plus more than 5% oxygen	Check for tightness of transformer.
3.	Nitrogen, Carbon dioxide and/or Carbon monoxide	Transformer overloaded or operating hot causing some cellulose breakdown. Check operating conditions.
4.	Nitrogen and Hydrogen	Corona discharge, electrolysis of water or rusting.
5.	Nitrogen, hydrogen, carbon dioxide, and carbon monoxide	Corona discharge involving cellulose or severe overloading of transformer.
6.	Nitrogen, hydrogen, methane with small amounts of ethane and ethylene.	Sparking or other minor fault causing some breakdown of oil.
7.	Nitrogen, hydrogen, methane with carbon dioxide, carbon monoxide and small amounts of other hydrocarbons. No acetylene.	Sparking or other minor fault in presence of cellulose.
8.	Nitrogen with high hydrogen and other hydrocarbons including acetylene.	High energy arc causing rapid deterioration of oil.
9.	Nitrogen with high hydrogen, methane high ethylene and some acetylene.	High temperature arcing of oil but in a confined area. Brazed connections or turn-to-turn shorts are examples.
10.	Same as (9) except carbon dioxide and carbon monoxide present.	Same as (9) except arcing in combination with cellulose.

It is recognized that there are many variables that prevent clear cut interpretation of faults found in transformers and the steps that must be taken to eliminate the gassing problem. However, in many cases the technique of monitoring and analyses of gases generated in transformers has become a valuable tool in the diagnosis of the transformer fault and how best to correct it. There is little doubt that improvements in testing procedures will provide new information to permit better maintenance techniques for transformers.

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I - Gas evolution

II - Detection and Interpretation

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B) Gas Cushion in Sealed Transformers

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