FLUIDS FOR USE IN DEEP ICE-CORE DRILLING

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Abstract: Environmentally appropriate fluids were sought to support the deep ice coring objectives of the glaciological programs of the U.S. National Science Foundation. In the past several decades, three types of fluids have been used for ice coring activities: 1) fuel oil (DFA) usually containing several percent of a dense halogenated solvent; 2) aqueous ethanol or glycol solutions; and 3) butyl acetate. Each has advantages and disadvantages.

Primary criteria for the search for acceptable ice coring fluids was that its density be close to that of the ice, that it be usable to about -56° C, and that it be as environmentally acceptable and non toxic as possible. The best candidate drilling fluid for ice coring that emerged was n-butyl acetate. This fluid is presently in use in Greenland.

Regardless of the fluid chosen, proper practices should be adopted for adequate protection of human health and the natural environment.

1. Introduction

In the past several decades, three types of fluids have been used for either ice drilling or coring operations: 1) fuel oil, usually containing several percent of a dense halogenated solvent, 2) aqueous ethanol or glycol solutions, and 3) butyl acetate. The first case is doubly objectionable to both the environment and to the workers in that it contains chlorinated substances and aromatic components, such as benzene. Aqueous ethanol is a reasonable to good choice but has two potential drawbacks (GOSINK *et al.*, 1991a). One is its lower flame limit, and the other is its propensity toward contaminating the subsurface layer of an ice core for parts per trillion (10^{-12}) range chemical analyses, which are now standard practice.

In addressing the problem of finding an acceptable alternative ice coring fluid, safe for the worker, the environment, and the ice core, the first step was to perform a computer search of the chemical literature. The initial primary criteria for that search were the density of the liquid and its freezing point. The desired density was that of ice (920 kg/m^3) and a freezing point at or below the worst-case scenario (-55°C) as might be encountered in operations in Antarctica. The initial search turned up a little over a hundred chemical names that fit the simple criteria. It was a simple matter to eliminate most of these in that they contained halogen, cyano, thio, acid, or equally objectionable functional groups. The second computer search of the remaining few dozen compounds was based on their known hazard assessments. Flammability and general toxicity factors trimmed the list to less than a dozen potential fluids. Ethanol was not in this list because it alone did not satisfy the density criteria. Only aqueous ethanol has sufficient density. Ethanol has been used in ice coring projects (ZOTIKOV, 1979; MOREV *et al.*, 1989; ZAGORODNOV, 1988, 1989) and can be considered for some types of coring operations. Further scrutiny of the remaining compounds yielded butyl acetate as the only truly viable alternative (GOSINK, 1989; GOSINK *et al.*, 1989, 1991b). Flammability, mild noxious properties, water solubility, viscosity, and ready commercial availability constituted the main final criteria. Recommendation of butyl acetate is also based on the fact that even numbered carbon chains (*e.g.*, butyl-4) are less toxic (narcotic) than the odd numbered (*e.g.*, propyl-3 or amyl-5), and it is more readily available.

Ethylene glycol solutions have been employed for several decades in deep ice drilling projects (UEDA and GARFIELD, 1969a) and aqueous ethanol for a decade or more (ZOTIKOV, 1979; ZAGORODNOV, 1988; MOREV *et al.*, 1989). Fuel oils containing small quantities of halogenated compounds (28:1) to increase the density of the mixture have also been employed in deep ice drilling projects for several decades (UEDA and GARFIELD, 1969b). There are indeed several desirable features for the use of aqueous ethanol as an ice drilling fluid (cost, environmental, and personal safety), but there are serious questions about its density and viscosity that the user must also consider. Glycol solutions become extremely viscous at low temperatures (HANSEN, 1976; UEDA and GARFIELD, 1969a) and present some density overturn problems if the bottom of the borehole is significantly warmer than the upper reaches of the hole. Aqueous ethanol is an improvement over ethylene glycol with respect to viscosity problems, is decidedly cheaper to employ than even fuel oils, and is safer for the workers and the environment. However, the potential for corrosion of the ice sample is greatest with alcohol, even at low temperatures if not properly monitored.

Personal health and fire safety for the worker, the scientist, and the remote facility are driving factors of concern no matter what the nature of the organic fluid used in ice coring or drilling operations. Permissible doses and flammability data are routinely available for most compounds. The problem of reliably detecting the vapors at the acceptable level is exacerbated by the low temperatures at which ice operations are carried out. While simple color reaction tube tests are routinely used in many commercial operations, some of them have severe temperature limitations. The detection tube designed for butyl acetate vapor detectors are also temperature dependent, becoming ineffective or unreliable at temperatures near 0°C. The device must be dependable at temperatures of -30°C. Calibration is also a challenge at these temperatures because of the penchant of the vapors in the container for the standard to condense.

Fortunately, several devices are available that can operate at these low temperatures, or by employment of precautions or thermal jackets, and can be relied upon to provide the necessary work-place monitoring data.

2. Discussion

2.1. N-butyl acetate as a drilling fluid

2.1.1. Density

The hydrostatic pressure, which is a function of density and depth, is of major importance in the selection of ice core drilling fluids. The density of butyl acetate increases with decreasing temperature (Fig. 1) and at temperatures below -15° C is sufficient, being greater than the firn-ice-layer density of 900 kg/m³ (PATTERSON, 1981).

At -30° C the butyl acetate is denser than pure ice. A 10% mixture of anisole in butyl acetate is as dense as pure ice at -15° C. Typically, the top of the fluid level in a borehole is about 100 m below the ice surface to balance the ice matrix pressure. Since the average internal temperature in the Greenland glaciers has been observed to be -31° C and is expected to be significantly colder in Antarctica, an added densifier is not required. If a densifier is required for temperate glacier drilling operations, anisole is much more desirable than PCE, TCE, or PBBE with respect to water pollution, human toxicity, and air pollution.



versus temperature: (a) anisole, (b) 10% anisole in butyl acetate, (c) butyl acetate, (d) 0.91 bromoil, (e) pure ice, 10% anisole in butyl acetate, (c) butyl acetate, (d) 0.91 bromoil, (e) pure ice, 10% acetate, (c) butyl acetate, (c) butyl

2.1.2. Viscosity

and (f) fuel oil.

The viscosity of the borehole fluid determines the travel time of the drill string, particularly at greater depths, and thus to the overall cost of the project. A low viscosity fluid must be employed. Figure 2 shows the effect of drill fluid viscosity on velocity of the fall of the PICO mechanical drill string employed in the Greenland Ice Sheet Project (GISP-2) (PROENZA *et al.*, 1990; RINALDI *et al.*, 1990). At 10 cp it is about 0.65 m/s; at 2 cp about 3.1 m/s. Figure 2 shows the round-trip time for the drill string going to 1000 m depth. It is calculated to be about 10 min with butyl acetate, but nearly an hour and a half with bromoil with its viscosity of about 15 cp at -30° C.

The general desire for the PICO mechanical drill is that the viscosity of the drill fluid be less than 5 cp. As can be seen in Fig. 3, the viscosity of butyl acetate (and the 10% anisole mixture) remains below 3 cp, even at -50° C, whereas the viscosity of bromoil is substantially above 15 cp. All bearings suffer at these low viscosities, but needle or roller bearings survive.

2.1.3. Volatility/Flammability

The greater volatility of butyl acetate raises fire safety questions. It is unlikely that under the conditions experienced in ice-core work that this will be a problem. There is confusion in the technical literature on the flash point of butyl acetate. SAX and LEWIS (1989) report 22°C. Another high-quality butyl acetate label quoted 33°C. The Merck Index (1976) quotes 38°C. Determination of the flash point by a local independent testing laboratory indicated 29.4°C, in keeping with our observations. Published safety data states that the lower flame limit concentration of n-butyl acetate in air is 1.7% (1988–89 CRC handbook). This corresponds to a vapor pressure of 12.9 mm Hg, achievable only in an unventilated closed room or container at 24°C. At -20° C, an average-to-cool day on the drill site, the maximum possible air concentration of butyl acetate in an unventilated area would be approximately 1300 ppm (0.13%), well below the lowest lower flame limit of 1.4%. At higher altitudes the lower flame limit is still in the +15°C to 18°C range. The warmest part of the drill site days, according to temperature records from past sites, rises to about 0°C. Odor detection and irritation both occur below the permissible exposure limit (150 ppm) and, thus, provide both adequate physiological and fire-warning properties (GOSINK and KELLY, 1991; GOSINK *et al.*, 1991b).

Side benefits of the greater volatility of butyl acetate are:

- 1) No oily residue will remain on work clothes between shifts of drilling crews.
- 2) Residual odor will disappear rapidly depending on degree of ventilation.
- 3) The core will be much less "greasy" to handle.
- 4) The lower viscosity and higher volatility should pose less of a hazard for making the driller's deck slippery.



Fig. 3. Viscosity of potential ice drilling fluids versus temperature: (a) 0.91 bromoil (LVT-200 + PBBE), (b) LVT-200, (c) 10% anisole in butyl acetate, and (d) butyl acetate.





2.1.4. Solubility

The results of several experiments, in which a 20-g cube of ice was placed in a covered beaker with about 100 ml of solvent, are shown in Fig. 4. The published solubility of butyl acetate in water is about an order of magnitude lower than ethyl acetate and is essentially the same as that for PER or about 0.7%. The solubility of water in butyl acetate is 1.6%. Note that the weight loss of ice in wet butyl acetate was not noticeable in seven hours contact time.

2.1.5. Solvent effect on polymers

The effect of butyl acetate and several other solvents on a variety of polymers is shown in Table 1. Both butyl acetate and anisole have no effect on the various polymeric materials employed in the PICO deep drill string. Recent inquiry about various epoxy resins and other plastics also show no deleterious effects. The wires in an electric motor, even at temperatures of 125°C are not affected by either butyl acetate or the 10% mixture of anisole. The PICO deep drill motor, which contains brushes, has recently been operated successfully at full power immersed in butyl acetate at 25°C for over 700 hours. The manufacturer stated that the tensile strength of the Kevlar cable used in PICO's drilling operations is not affected by butyl acetate (letter from Cortland Co.). However, the proprietary lubricant on the Kevlar cable washed out causing weak spots and making it necessary to abandon the cable after three seasons in the field. Unlubricated cross-woven Kevlar fibers appear to cut each other after many trips over a sheave. Butyl resistant lubricants and a different wrap configuration may solve this problem.

Plastic	No. 1 Fuel oil	0.91 Bromoil	PER	LVT-200	Anisole	n-Butyl acetate
Black rubber	SS-	ss+	ss+	se+	se	se
Polypropylene	ss–	SS	ne	se	ne	ne-
Teflon	ne	ne	ne	ne	ne	ne
Viton	ne	ne	ne	ne	ne	ne
Elec. insu.*		ne	ne		ne	ne
Elec. insul. [†]						ne
Polyethtylene						ne
Kevlar						ne
Nylon						ne

Table 1. Solvent effect of the butyl acetate on polymers (24 + 3 hrs).

se = slight effect ss = soften and/or swell

+ = more - = less-*i.e.*, a very slight effect or softening

ne = no effect (did not swell, soften or become tacky)

• Lacquer on wire, 125°C for 2 hrs.

* Plastic jacket on wire.

A blank area indicates no data.

2.1.6. Trace elements

A recent analysis of a grade ($\geq 99.5\%$) of butyl acetate indicated contaminant concentrations of: < 0.01% water, 0.07% butanol, and < 0.01% acetic acid. An analysis based on a 1:1 water extraction was reported as: chloride, < 0.5 ppm; sulfate < 0.1 ppm, *i.e.*, below the routine detection limits of the analyzer. Trace metals in butyl acetate from a test on a drill string were on the order of 1 or 2 ppm, and with iron (probably from the container) about 20 ppm. The presence of 1 ppm of drill fluid would thus contaminate the ice at the part per trillion level.

2.1.7. Indoor ambient air quality

Butyl acetate odors are noticeable at 10 ppm. At approximately 100 ppm ambient concentration, some nasal irritation may be noticed. This is well below the Occupational Health and Safety Administration's (OSHA) limit of 710 mg/m³. Kerosene fumes are only

permitted to be 100 mg/m^3 of air. Therefore, before any significant health hazard would be present, odors of butyl acetate would be noticeable and irritating.

Potential problems of excessive butyl acetate concentrations in the air can be eliminated by proper design and use of ventilation systems in the drill enclosure, core relaxation pit, and science trench. Fans to keep the interior core handling structures cold should be part of the management plan and will automatically ensure that butyl acetate vapors will be kept to an acceptable minimum.

2.1.8. Health hazards

Butyl acetate is slightly hazardous to health. It is a mammalian reproductive toxin. The main risk, however, comes from prolonged inhalation of vapors in excessive concentrations. Butyl acetate is not listed as a carcinogen as are PBBE and TCE. Several inexpensive battery- or a.c.-operated alarms are available at low to moderate cost. These alarms are sensitive enough to warn at the parts per million level for health purposes, or at higher ranges for fire danger warning, and operate at -5° C without the need of a warmed housing (GOSINK and KELLEY, 1991).

2.1.9. Environmental characteristics

From an environmental aspect, butyl acetate and other liquids such as ethanol are easily biodegradable compounds as opposed to the mixture of components in fuel oil, LVT-200, or PBBE. Both fuel oils and LVT-200 pose higher environmental risks since, in the event of a spill (or the long-term eventual release of residual material left in the glacier), they have a long residence time, particularly in a cold environment (Fig. 5). In a worst-case scenario, if all the butyl acetate leaked out the bottom and flowed into a fjord, most of it should evaporate in one to three days. An additional small amount would disperse in the water column, where it would be biodegraded. Butyl acetate will, contrary to all the other fluids proposed or in use, totally clean itself up in a matter of days, even in a cold climate.

2.2. Ethanol as a drilling fluid

We have examined the available literature and conducted laboratory and field tests to ascertain whether or not aqueous ethanol could be used as a reliable drilling fluid for retrieval of glacier ice cores for chemical and physical analyses.

2.2.1. Corrosion

Ice cores from 130-m depth at Summit, Greenland, were immersed in red dyed aqueous ethanol (26%) at equilibrium conditions for 15 minutes and for over two hours (Cole-Parmer, part number N-00298-05). Both core samples were pink in color when removed from the deeply dyed aqueous ethanol bath, but became colorless to the unaided eye when they were rinsed with more of the undyed solvent, also at equilibrium conditions. Tool marks (0.5 mm deep) were still visible, but fracture marks on the top of the ice were rounded after 15 minutes' contact. No pitting was noted on the side of the short-term exposure core. The core exposed over 2 h to equilibrium aqueous ethanol shows significant rounding of the tool marks and slight pitting. In addition, slight, irregularly spaced penetration of the dye was observed. The dyed spots appear to be less than 2 mm in depth and length.

2.2.2. Penetration

Figure 6 shows the penetration of aqueous ethanol in samples of Greenland ice. Trace



Fig. 5. Evaporation of drilling fluids (at 22°C) versus time (5 ml of fluid in a 50 ml beaker):
(a) butyl acetate, (b) anisole, (c) butyl acetate at -18°C, (d) fuel oil, and (e) LVT-200.



Fig. 6. Penetration of ice core by drilling fluids (a) 26% ethanol at -15°C after 15 min contact;
(b) 50% ethanol at -31°C at 2 bar pressure in borehole; and (c) pure butyl acetate at -31°C.

"a" shows the data obtained from the sample treated with dyed ethanol (26%) at -15° C for 15 min in a cold room at Summit (GISP-2), Greenland. In another experiment, aqueous ethanol (50% by volume) was introduced to the 150-m depth borehole at Summit, Greenland, at the end of the drilling season. The level of the fluid was about 20 m. A short core was obtained and returned to this laboratory for analysis. Trace "b" represents data obtained from the sample drilled under 2 bar pressure of an equilibrium concentration of ethanol at -31°C. About three months later, the cold-stored cores were lathed and the melted shavings analyzed. Other samples were scraped from these test cores at an earlier date and showed essentially the same results. Analysis of the ethanol in the melted ice scrapings was accomplished by flame ionization detection gas chromatography. The subsample from the outer half-millimeter surface of the dye-exposed ice revealed a slight pink color. Samples from deeper in the ice were colorless. Chemical analysis revealed that most of the ethanol (≥ 10000 ppm) remained in the outer 1 to 2 mm of the core, but that chemically significant penetration occurred to 15-mm depth. Ethanol was not detected in any of the samples from the center 60-mm diameter portions of all the ice samples. Replicate analyses show the error to be about + 3% at the 10 ppm level; + 20% at and below the 1 ppm level. The detection limit for the method is 0.2 ppm. The level, or stair step, in the penetration curves is real, having been observed in several separate samples and separate scraping or lathing experiments. Penetration by hydrophobic butyl acetate is also shown in Fig. 6c. The concentration of the entrained butyl acetate is one to two orders of magnitude lower than the results for alcohol.

2.2.3. Density

Figure 7 presents density for aqueous ethanol solutions at or very near their freezing points. CRC handbook data for the density at 20°C of aqueous ethanol is provided for comparison purposes. The straight line labeled "pure ice" is an extrapolation of the 0°C to -30° C data by PATTERSON (1981). At -51° C the density of 72% ethanol in water appears to be slightly less than that of ice. Internal glacier temperatures near -50° C are anticipated for drilling projects in Antarctica, at least in the upper portions of the glacier, particularly

in the East Antarctic plateau. Elsewhere in Antarctica internal temperatures may vary from approximately -20° C to -35° C. The required less dense fluid (72% ethanol in water) would be stable on top of the more dense solutions that would be introduced to the deeper portions of the borehole.

The aqueous ethanol densities are of greater density than butyl acetate or hydrocarbon mixtures so that shavings from the mechanical coring operation may rise more rapidly, thus increasing the likelihood of slush jamming the upper portion of the hole.



2.2.4. Viscosity

The viscosity data presented in Fig. 8 is for an aqueous ethanol solution at or very near its freezing points. The percent of ethanol content is indicated by the numbers at the top of the figure. For the PICO mechanical drill, it is desirable that the viscosity of ice core drilling fluids be 5 cp or less. A discussion of this time and energy point is presented in GOSINK *et al.* (1991b). In brief, it will take the drill string 10 min for one round trip in a 1000-m hole where the viscosity or the drill fluid is 3 cp compared to 80 or 90 min if the viscosity is 15 cp. Fluids with viscosity of no more than 20–25 cp are workable in moderate depth holes (≤ 1000 m). While the prospect of 50 cp fluid in Antarctic operations is not desirable, it can be overcome and is substantially better than the extremely adverse viscosity problems associated with ethylene glycol solution. For example, HANSEN (1976) used terms such as "pasty" or "difficult to work" for 20 to 50% ethylene glycol-water mixtures. The low viscosity (< 5 cp) is a serious problem for ball bearing lubrication, but not for roller or needle bearings. A viscosity closer to 20 or 25 cp is desirable if sealed bearing chambers must be avoided.

2.2.5. Safety-Personal, environmental, and fire

Ethanol is the best of all fluids even over our previously recommended butyl acetate from an environmental and personal safety consideration. In the work place air, 1000 ppm of the alcohol vapors are permitted. (10000 ppm for several hours will induce intoxication in some people and is near the fire limit.)

As far as the environment is concerned, ethanol is a natural, widely occurring product.

Ethanol would be rapidly consumed by the microbiota in any water body into which it might be spilled, and its infinite solubility would assure its dispersion.

The volatility of ethanol is good in terms of a land spill, but leads to some concern with respect to fire hazard. Its flash point is lower than that of butyl acetate, which is considered safe only in view of the low temperatures at which it will be utilized. The same is still marginally true for cold aqueous ethanol. It should be noted that ethanol has been used safely as a drilling fluid for many years. There is one mitigating factor, and that is that only 20–70% solutions of ethanol will be employed. Table 2 shows the flash point of ethanol and aqueous ethanol. The data were determined in a commercial testing laboratory or taken from the literature. Butyl acetate and fuel oil data are added for comparison.

Percent alcohol	Flash point (°C)	19-1
100	10	
50	22	
30	28	
(Butyl acetate)	29	
(Fuel oil)	~ 66	

Table 2. Measured flashpoint of ethanol aqueous solutions (in comparison with other fluids).

2.2.6. Cost

By using ethanol, the potential cost saving amounts to many tens of thousands of dollars per bore hole. The major cost advantage comes not from the purchase, but in the transportation cost to the remote drill sites (about \$3 to \$4 per kg). Nominally, 50% solutions would be required for deep drilling in West Antarctica, and the snow will not require any (or very little) added heat because of the corrosive nature of ethanol towards snow.

2.2.7. Miscellaneous

The ethanol available from commercial manufacture (Quantex Corporation) is totally synthetic, derived from petroleum, thus eliminating any C-14 problems. While pure water (glacier ice) and ethanol are not good electrical conductors in themselves, one will have to employ sealed electrical motors and electrical connections to ensure against damage to the motors. Metal corrosion is also more likely in protic solvents.

2.3. Health considerations

Our attraction to hostile environments and remote areas historically has been driven by the quest for adventure, scientific interest, and exploration. The hardships endured by early explorers were at one time considered part of the wilderness experience. In polar regions frostbite injury became the accepted "badge of courage". These regions were reserved for the robust. Today, however, our technology affords us greater safety and comfort in extreme environments and permits increased human activity. The "world's expert", who may be a senior staff member and less hardy than earlier explorers, is now able to bring his expertise on-site. Present day explorers of oceans, polar regions, and space are workers. Workers are often employed as scientists, technicians, riggers, and construction and support personnel. For them, it is work as usual despite location. The hardships of the wilderness experience must be minimized for these workers, and accepted practices and regulation of health and safety in the work place applied.

Although work sites may be outside the national boundaries, employers based in the United States are subject to Occupational Safety and Health Administration (OSHA) standards and state compensation boards. It is incumbent upon employers by regulation to afford workers adequate protection from hazards in the work place, conduct medical surveillance, and provide medical treatment and compensation for injuries incurred at work. Today, the usual concerns of employee exposure to toxic substances, excessive noise, and risk of traumatic injury have been expanded to include blood-born pathogen exposure. The latter issue greatly complicates incident reporting procedures and medical response to injury, especially if definitive care occurs outside the U.S., and blood products are required in treatment of the victim.

The necessity of workers residing in intimate association with the job site work and each other, essentially closed habitat living, for prolonged periods enhances opportunity for industrial exposures as well as transmission of communicable illness. The boundaries between work place and residence become blurred.

PICO has addressed many of these issues by adherence to OSHA standards of respiratory protection of workers handling drilling fluids and periodic medical surveillance practices. In addition, a medical facility is present on-site staffed by a physician who is able to provide primary care. Emergency evacuation plans have been established. Detailed medical records are maintained and state compensation board reporting practices are followed. Staff training and a sound safety program are required to prevent serious injury at the site. Employers should follow standard practices of health and safety for their workers.

3. Summary and Conclusions

Butyl acetate represents a significantly lower risk to the environment and to the worker than other fluids such as fuel oil with densifiers. Fuel oil also is resistant to environmental degradation and subsequently has a long residence time in the environment. Persistence of these hydrocarbon compounds and their densifier components is increased in cold temperatures. In the event of a spill, or in the inevitable long-term release of residual material left in the glacier, the persistence of these compounds will represent an environmental hazard. In contrast, butyl acetate is relatively easily degraded in the environment. Furthermore, any unexpected releases or spills should evaporate quickly and present little environmental or safety hazard.

The fire hazard of butyl acetate is very low at temperatures below about 0 to 10°C and, thus, does not pose any inordinate danger.

Contamination of the ice core by butyl acetate appears to be acceptably low for most chemical and physical measurements.

Aqueous ethanol may be a useful ice core drilling fluid in warm ($\geq 25^{\circ}$ C) bore holes and where density overturn is not a problem, that is, in holes of moderate depth. The viscosity properties of aqueous ethanol are vastly superior to those of ethylene glycol solutions, even at -55°C, and will not be significant for shallow to moderate depth (< 1000 m) bore holes. Definitive tests will have to be performed on trace metal problems because of potential minor corrosion of the drill string by ethanol and by water. Sealed motors and potted electrical connections will have to be employed in electromechanical drill strings. Further tests with mechanical drilling systems are necessary to prove the concept since the bulk of drilling experience is with thermal drills.

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