Dr. C. C. Kitchens
August 27, 1951

1. Prod. Dir.
2. Dr. Kitchens - 8/27/51
3. destroyed 9/28/70
4.
5. destroyed 9/28/70

Recovery of G-11 from Sediment

in Sewer Ditch

Project No. 97.

Introduction:

Sediment has collected in the pond, which was a part of our old sewer system. Individual samples of the sediment from various locations in the pond as well as representative samples of the whole pond have been collected and found to consist of principally G-11 and inorganic matter (sand, filtercel, grit and calcium salts).

Summary:

Representative samples of the sediment yields 60% G-11 Pure (based on the dry solid content of the sediment). The G-11 content is not uniform over the whole pond, the solids near the west end of the pond analyzes about 70% G-11, while the G-11 content near the middle and east end of the pond is about 50% G-11. The sediment from the middle and east end of the pond contains, besides G-11 and inorganic materials, about 5% of oily residue which is easily removed.

The G-11 is easily recovered as G-11 Pure by solvent or caustic extraction followed by crystallization from solvents. The best method for plant operation was found to be a slight modification of the G-11 Pure process.

GIV_NBC_0664662

Recovery Procedure:

Two hundred fifty grams (43.5% solids) of a representative sample of the sediment was agitated 30 minutes at 40°C with 620 g. of 12.5% NaOH solution. 5 g. of filtercel was added and the batch was filtered giving an insoluble residue precipitate of 30 g.

The caustic solution of G-11 was extracted with 100 ml. of benzene to remove any insoluble oily material. Evaporation of the benzene extract gave 3 g. of oily residue. The sodium salt of G-11 was precipitated from the caustic solution by adjusting the pH to between 10 and 10.3 with 62½% H_2SO_4. The batch was agitated 30 minutes and the mono sodium salt of G-11 was filtered and washed with 10 ml. of water.

The caustic solution, freed of G-11, gave 8 g. of acidic materials on acidification with 62½% sulfuric acid.

G-11 was isolated from the sodium salt by suspending the salt in 200 ml. of water and acidifying to congo paper with 62½% sulfuric acid. The G-11 was filtered and washed with water. This G-11 had a good melting point, however, the color and alcohol solubility was poor.

To obtain pure G-11, the wet G-11 was added to 150 ml. of toluene and refluxed while removing the water azeotropically. 10 g. of superfiltrol was added and after refluxing 15 minutes, the toluene solution was filtered and allowed to crystallize. There was obtained 58 g. of G-11 Pure. Evaporation of the mother liquor to 50 ml.

and crystallizing gave an additional 5 g. of pure G-11. The removal of the solvent from the second mother liquor gave a residue of 6 g. of impure G-11.

The total yield of pure G-11 was 63 g. or 58%;

M.P. 163 - 164°C.
Color White
Alcohol Solubility - Clear.

DATA

SAMPLE LOCATION	% Moisture	% Based on Solid Sediment		
		% G-11	% NaOH insol.	% NaOH soluble at pH 10
West end	15.3%	73.5%	26.1%	trace
Center (west end)	18.6%	70.0%	26.0%	3.0%
Middle	68.8%	53.0%	23.0%	10.2%
Bottom (near middle)	50.2%	45.0%	47.0%	5.0%
South side	34.5%	51.0%	31.0%	6.4%
East end	39.6%	23.9%	20.6%	10.0%
Representative	44.7%	60.1%	31.2%	4.8%
Representative	56.5%	58.0%	22.9%	7.4%

Conclusion:

The solid sediment will yield about 60% pure G-11. The exact estimate of the total amount of G-11 available from the sediment is difficult because of the nonuniformity of the G-11 and moisture contents, however, a conservative estimate of 10,000 lbs. or more is reasonable.

Garry C Kitchens

Dr. G. C. Kitchens,
Delawanna, N. J.,
August 27, 1951.