10/29/82

DYNAMAC
CORPORATION

NALED

Product Chemistry

Task 1: Review and Evaluation of Individual Studies

Contract No. 68-01-5830

Final Report

October 29, 1982

Submitted to:
Environmental Protection Agency
Arlington, Virginia 22202

Submitted by:
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Enviro Control Division
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NALED

PRODUCT CHEMISTRY

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CASE GS 0092 NALED STUDY 1 12/22/81 PM 110

CHEM 034401 Naled

BRANCH RCBR DISC 05 TOPIC 05 GUIDELINE 40 CFR 163.61-8

FORMULATION 00 - ACTIVE INGREDIENT

FICHE/MASTER ID 00074653 CONTENT CAT 12

Chevron Chemical Company (1966) Name, Chemical Identity and Composition of the Pesticide Chemical: Dibrom. (Unpublished study received Sep. 12, 1966 under 7F0532; CDL:092821-H).

FICHE/MASTER ID 00065493 CONTENT CAT 12

Chevron Chemical Company (19??) Composition of Technical Naled. (Unpublished study received Mar. 19, 1976 under 239-2444; CDL:229289-F).

FICHE/MASTER ID 00065494 CONTENT CAT 06

Hayman, E.L.; Friedrich, W.E.; Carlstrom, A.A. (1971) Determination of Impurities in Technical Dibrom. (Unpublished study received Mar. 19, 1976 under 239-2444; submitted by Chevron Chemical Co., Richmond, Calif.; CDL:229289-H).

SUBST. CLASS = S.

DIRECT RVW TIME = 5 (MH) START-DATE May 7 END DATE May 10

REVIEWED BY: N. Hajjar
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SIGNATURE: Nicolas P. Hajjar DATE: May 10, 1982

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Conclusions:

Three confidential business documents submitted by Chevron Chemical Co. provide information on the composition of technical naled. The available data partially satisfy data requirements in Sections 163.61-5 and 163.61-6 of EPA's Proposed Guidelines for Registering Pesticides (July 1978) by providing the identity of potential impurities. However, data gaps include: i) unacceptable or inadequately described methods; ii) sensitivities and detection limits were not reported for any of the methods used; iii) no indication was made as to whether samples of technical naled were from different batches; iv) early and later documents report two different naled limits; v) several impurities that could be present at ≥0.1% (by weight of technical) were not identified or quantified individually; and vi) certification of limits was not made.

Methods and Materials:

[redacted]

Reported Results: QUALITY CONTROL PROCEDURE INFORMATION IS NOT INCLUDED

An early document of Chevron Chemical Co. (1966, 00074653) showed that 10 samples of technical naled (source: Shell Chemical Co.) contained the active ingredient at [redacted] Later documents (Chevron Chemical Co., 19??, 00065493 and 1971, 00065494) reported the limits as [redacted] The impurities in technical naled were reported.

Discussion:

The available data only partially satisfy data requirements (identity of potential impurities) because: i) all methods were either unsuitable or inadequately described; ii) sensitivities and detection limits were not reported for any of the methods used; iii) there was no indication that samples of technical naled were from different batches; iv) there is a discrepancy between early and later reports regarding the naled limits; and v) several impurities that could be present at ≥0.1% (by weight of technical) were not identified or quantified individually.

CASE GS 0092 NALED STUDY 2 12/22/81 PM 110

CHEM 034401 Naled

BRANCH RCBR DISC 05 TOPIC 05 GUIDELINE 40 CFR 163.61-8

FORMULATION 00 - ACTIVE INGREDIENT

FICHE/MASTER ID 00074653 CONTENT CAT 12

Chevron Chemical Company (1966) Name, Chemical Identity and Composition of the Pesticide Chemical: Dibrom. (Unpublished study received Sep. 12, 1966 under 7F0532; CDL: 092821-H).

FICHE/MASTER ID 00074791 CONTENT CAT 06

Chemical Chemical Company (19??) Naled (1,2-Dibromo-2,2-dichloroethyl Dimethyl Phosphate): Manufacturing Process. (Unpublished study received Oct. 17, 1977 under 239-1633; CDL: 232095).

SUBST. CLASS = S.

DIRECT RVW TIME = 1 (MH) START-DATE May 7 END DATE May 7

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Conclusions:

Two confidential business documents submitted by Chevron Chemical Co. (00074653 and 00074791) describe the process used to manufacture technical naled. Purities of starting materials are provided. The available information satisfies data requirements in Section 163.161-4 of EPA's Proposed Guidelines for Registering Pesticides (July 1978).

Methods and Materials:

The procedures used to manufacture technical naled, including the purity of certain starting materials, are presented.

Reported Results:

N/A.

Discussion:

N/A.

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CHEM 034401 Naled

BRANCH RCBR DISC 05 TOPIC 1015 GUIDELINE 40 CFR 163.61-7

FORMULATION 01 - TECHNICAL CHEMICAL

FICHE/MASTER ID GS092006 CONTENT CAT --

Carlstrom, A.A. 1975. Gas-liquid chromatographic determination of naled in pesticide formulations. JAOAC 58(6):1162-1168.

FICHE/MASTER ID 00074846 CONTENT CAT --

Chevron Chemical Company. 1964. Analysis of Dibrom. Method [D-IV] dated July 21, 1964. Unpublished study received June 23, 1965 under unknown admin. no.; CDL:102845-A.

FICHE/MASTER ID 00074655 CONTENT CAT 06

Chevron Chemical Company. 1966. Ortho method of analysis--D-IX-a: Dibrom (R) Naled by gas chromatograph. Method dated May 27, 1966. Unpublished study received Sep. 12, 1966 under 7F0532; CDL:092821-J.

FICHE/MASTER ID 00074724 CONTENT CAT 06

Ospension, N.J. 1958. Letter sent to G.K. Kohn dated Feb. 4, 1958: Dibrom--physical and chemical properties. Includes method dated Apr. 3, 1957. Unpublished study received Feb. 10, 1958 under unknown admin. no.; submitted by Chevron Chemical Co., Richmond, Calif.; CDL: 119717-A.

SUBST. CLASS = S.

DIRECT RVW TIME = 4 (MH) START-DATE May 10 END DATE May 10

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Conclusions:

Three Chevron Chemical Co. documents and one published study describe analytical methods for the determination of naled in technical and formulated products. A method involving the potentiometric titration of Br^- is inadequate because it is not specific for naled (00074724). The GC methods (00074655, 00074846, and GS092006) also do not satisfy data requirements in Section 163.61-7 of EPA's Proposed Guidelines for Registering Pesticides (July 1978) for the following reasons: detection limits and validation data were not provided, data for technical naled or formulated products were not reported, and a confirmatory procedure was not detailed. An update of the method(s) used for quality control of the technical and formulated products of naled is required.

Methods and Materials:

Br^- titration method QUALITY CONTROL PROCEDURE INFORMATION IS NOT INCLUDED

Chevron Chemical Co. (Ospenson, 1958, 00074724) [redacted]

GC Method D-IX-a (Chevron Chemical Co., 1966, 00074655)

[redacted]

GC Method D-IV (Chevron Chemical Co., 1964, 00074846)

[redacted]

AOAC GC method, 1975, GS092006 QUALITY CONTROL PROCEDURE INFORMATION IS NOT INCLUDED

Chloroform-soluble material in samples of technical naled and formulated products is analyzed by using a GC equipped with a hydrogen flame detector. A column containing 3% XE-60 on Gas-Chrom Q is operated at 135°C. Pretreatment by repeated injections of the naled-dibutyl phthalate standard is required. Several pesticides were tested to determine if they interfere with naled determinations.

Reported Results:

The recovery of the Br^- titration method was 80%; the detection limit was not reported. There was also an indication that homogeneous solutions of naled were not obtained.

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Detection limits, validation data, and analytical data were not provided for any of the GC methods. It was suggested that typical peak shapes may not be reliably obtained by using Method D-IX-a. The type of detector used for Method D- IV was not reported. Benzene hexachloride, diazinon, and dicrotophos interfere with either naled or dibutyl phthalate using the AOAC method.

Discussion:

N/A.

CASE GS0092 NALED STUDY 4 PM 110 12/22/8

CHEM 034401 Naled

BRANCH RCBR DISC 05 TOPIC 05 GUIDELINE 40 CFR 163.61-8

FORMULATION 01 - TECHNICAL CHEMICAL

FICHE/MASTER ID 00074790 CONTENT CAT 12

Chevron Chemical Company. 1965? Product chemistry data for Chevron naled technical. Unpublished study received Oct. 17, 1977 under 239-1633; CDL:232095-A.

FICHE/MASTER ID 00074653 CONTENT CAT 12

Chevron Chemical Company. 1966. Name, chemical identity, and composition of the pesticide chemical: Dibrom. Unpublished study received Sep. 12, 1966 under 7F0532; CDL:092821-H.

FICHE/MASTER ID GS092040 CONTENT CAT --

Chevron Chemical Company. 1974. Chevron-Naled: Formulator's manual. Unpublished study received Oct. 22, 1974 under 239-1633, Accession No. 233083.

FICHE/MASTER ID 00074724 CONTENT CAT 06

Ospenson, J.N. 1958. Letter sent to G.K. Kohn dated Feb. 4, 1958: Dibrom--physical and chemical properties. Includes method dated Apr. 3, 1957. Unpublished study received Feb. 10, 1958 under unknown admin. no.; submitted by Chevron Chemical Co., Richmond, Calif.; CDL:119717-A.

SUBST. CLASS = S.

DIRECT RVW TIME = 10 (MH) START-DATE May 6 END DATE May 7

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Conclusions:

Four documents submitted by Chevron Chemical Co. (00074635, 00074724, 00074790, and GS092040) provide the names, identity, and physical/chemical properties of technical and pure naled. The information pertaining to technical naled partially satisfies data requirements in Sections 163.61-3 and 163.61-8 of EPA's Proposed Guidelines for Registering Pesticides (July 1978).

Methods and Materials:

N/A.

Reported Results:

The names, identity, and physical/chemical properties of technical naled, and in some cases pure naled, are presented below.

Name, identity, and physical/chemical properties of naled.

Chemical name:[a,b,c] 1,2-Dibromo-2,2-dichloroethyl dimethyl phosphate.

Common name:[a,b,c] Naled.

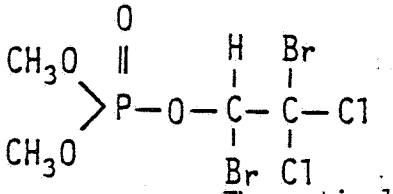
Trade name:[a,b,c] Dibrom.

U.S. Patent Number:[b,c] 2,971,882

Empirical formula:[a,b,c] $C_4H_7O_4PBr_2Cl_2$

Molecular weight:[b,c,d] 381

Structural formula:[a,b,c,d]



Molecular composition (% by weight):[d]	Theoretical	Found
H	1.84	1.98
P	8.13	8.30
C	12.62	12.63
Cl	18.62	18.30
Br	40.0	40.9
Total halogen	10.5 meq/g	10.49 meq/g

Melting point (°C): 25.56[c] (27-28.5[a,b], 25.5-26.5[d] for pure compound).

Boiling point (°C): 110[c] or 120[a,b] at 0.5 mmHg (108-110 at 0.25 mmHg for pure compound[d]).

Density/specific gravity (temperature not provided unless specified): 1.97 at 20°C[c]; 1.96[a,b]; 1.465[a] (1.96 for pure compound[d]).

Vapor pressure:[a,b,c] 2×10^{-4} mmHg at 20°C.

Refractive index:	n_D^{28} = 1.5108[b,d] or ∿1.512[c].
Infrared absorption:[b]	Not reported for technical (the pure compound absorbed strongly at ∿860, 900, 1020, 1060, 1120, 1280, and 1295 cm^{-1} as determined from the presented spectrum).
Viscosity:	22 cp at 20°C[a] or 210-250 sec Saybolt at 37.8°C[c].
Physical state:[a,c]	Oily liquid (pure compound is a low melting point solid).
Color:[a,c]	Light, straw-colored (white when pure).
Odor:[a,c]	Slightly pungent.
Miscibility:[a,c]	Miscible in most organic solvents; emulsifiable in water.
Solubility:[a,b,c]	Limited solubility (1-5%) in aliphatic solvents; highly soluble in oxygenated solvents such as ketones and alcohols; low solubility in water (0.2 g/100 ml at 23.3°C[a]).
Stability:	Saturated aqueous solutions of technical naled (2 g/l) are rapidly hydrolyzed at 23.3°C[a] (naled of unspecified purity in aqueous solution is not hydrolyzed at 0°C and has half-lives of ∿13 and ∿2 days at 25 and 40°C, respectively[b,d] complete hydrolysis occurs under reflux within 4 hours in distilled water or in the presence of excess NaOH[d]).
Flashpoint:[a]	53°C.
Dissociation constants:[a]	N/A (according to Chevron Chemical Co.).
pH:[a]	N/A (according to Chevron Chemical Co.).
Explosiveness:[a]	Not explosive.
Corrosiveness:	Corrosive to steel, aluminum, and magnesium[a]; corrosive to all the above as well as copper and brass when in the presence of water[c].
Oxidizing/reducing action:[a]	Not an oxidizing or reducing agent.
Storage stability:[b]	No breakdown of the technical material (source: Shell Chemical Co.) occurred within 18 months at ambient temperatures and 2.5% degradation occurred at 38°C over the same period.

[a]From 00074790.
[b]From 00074653.
[c]From GS092040.
[d]From 00074724.

Discussion:

N/A.